

STANDARD METHODS OF TESTING AND SPECIFICATIONS FOR CEMENT

CONTENTS

United States Government Specification for Portland
Cement.

Standard Specifications for Cement of the American
Society for Testing Materials.

Methods for Testing Cement—Report of the Committee
of the American Society of Civil Engineers.

Chemical Analyses.

ASSOCIATION OF AMERICAN
PORTLAND CEMENT MANUFACTURERS
BELLEVUE COURT BUILDING, PHILADELPHIA, PA.



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UNITED STATES GOVERNMENT SPECIFICATION FOR
PORTLAND CEMENT

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EXECUTIVE ORDER

It is hereby ordered that all Portland cement that may hereafter be purchased by any Department, Bureau, Office, or independent establishment of the Government, or that may be used in construction work connected with any of the aforesaid branches of the Government service, shall conform in every respect to the specification for Portland cement adopted by the Departmental Conference at the meeting held at the Bureau of Standards on February 13, 1912, and approved by the heads of the several departments (to be known as the United States Government Specification for Portland Cement): Provided, however, that such specification may be modified from time to time by any similar Departmental conference, with the approval of the heads of the several Departments.

WM. H. TAFT.

THE WHITE HOUSE,
April 30, 1912.

INTRODUCTORY STATEMENT

In June, 1911, the Secretary of the Department of Commerce and Labor arranged, through the Secretaries of the various departments, for a conference of Government engineers for the purpose of unifying the specifications for Portland cement used by the United States Government. At this conference a committee was appointed to consider existing specifications and to recommend a single specification for Portland cement to be used by all departments of the Government.

After an extended series of meetings of this committee, at which careful consideration was given to representative specifications for Portland cement, as well as to all available data on methods of tests, a tentative specification was developed, which was reported to the departmental conference at a general meeting held July 20, 1911.

In view of the desirability of agreement between the specifications in use by the public and those adopted by the Government, the committee was instructed by the conference to confer with representative consumers and manufacturers as well as the special committees of the national engineering societies more directly interested in the subject of cement specifications. As a result the tentative specifications first reported by the departmental committee were modified slightly, until substantial agreement was reached on practically all points except the methods of determining the normal consistency and time of setting, and the following specifications were unanimously adopted by the departmental conference at the meeting held February 13, 1912.

It was recognized that no specification can be considered final, but must be subject to revision from time to time as occasion requires, and provision will be made for such revision by the various Government departments, all of which have adopted the specification as recommended by the conference.

S. W. STRATTON,
Director.

Approved:
CHARLES NAGEL,
Secretary.

UNITED STATES GOVERNMENT SPECIFICATION FOR PORTLAND CEMENT*

I. SPECIFICATION

Definition.—1. The cement shall be the product obtained by finely pulverizing clinker produced by calcining to incipient fusion, an intimate mixture of properly proportioned argillaceous and calcareous substances, with only such additions subsequent to calcining as may be necessary to control certain properties. Such additions shall not exceed 3 per cent, by weight, of the calcined product.

Composition.—2. In the finished cement, the following limits shall not be exceeded:

	Per cent
Loss on ignition for 15 minutes.....	4
Insoluble residue.....	1
Sulphuric anhydride (SO ₃).....	1.75
Magnesia (MgO).....	4

Specific Gravity.—3. The specific gravity of the cement shall be not less than 3.10. Should the cement as received fall below this requirement, a second test may be made upon a sample heated for 30 minutes at a very dull red heat.

Fineness.—4. Ninety-two per cent of the cement, by weight, shall pass through the No. 100 sieve, and 75 per cent shall pass through the No. 200 sieve.

Soundness.—5. Pats of neat cement prepared and treated as hereinafter prescribed shall remain firm and hard and show no sign of distortion, checking, cracking, or disintegrating. If the cement fails to meet the prescribed steaming test, the cement may be rejected or the steaming test repeated after seven or more days at the option of the engineer.

Time of Setting.—6. The cement shall not acquire its initial set in less than 45 minutes and must have acquired its final set within 10 hours.

* Circular No. 33, Bureau of Standards, May 1, 1912.

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Tensile Strength.—7. Briquettes made of neat cement, after being kept in moist air for 24 hours and the rest of the time in water, shall develop tensile strength per square inch as follows:

	Pounds
After 7 days.....	500
After 28 days.....	600

8. Briquettes made up of 1 part cement and 3 parts standard Ottawa sand, by weight, shall develop tensile strength per square inch as follows:

	Pounds
After 7 days.....	200
After 28 days.....	275

9. The average of the tensile strengths developed at each age by the briquettes in any set made from one sample is to be considered the strength of the sample at that age, excluding any results that are manifestly faulty.

10. The average strength of the sand mortar briquettes at 28 days shall show an increase over the average strength at 7 days.

Brand.—11. Bids for furnishing cement or for doing work in which cement is to be used shall state the brand of cement proposed to be furnished and the mill at which made. The right is reserved to reject any cement which has not established itself as a high-grade Portland cement, and has not been made by the same mill for two years and given satisfaction in use for at least one year under climatic and other conditions at least equal in severity to those of the work proposed.

Packages.—12. The cement shall be delivered in sacks, barrels, or other suitable packages (to be specified by the engineer), and shall be dry and free from lumps. Each package shall be plainly labeled with the name of the brand and of the manufacturer.

13. A sack of cement shall contain 94 pounds net. A barrel shall contain 376 pounds net. Any package that is short weight or broken or that contains damaged cement may be rejected, or accepted as a fractional package, at the option of the engineer.

Inspection.—14. The cement shall be tested in accordance with the standard methods hereinafter prescribed. In general the cement will be inspected and tested after delivery, but partial or complete inspection at the mill may be called for in the specifications or contract. Tests may be made to determine the chemical composition, specific gravity, fineness, soundness, time of setting, and tensile strength, and a cement may be rejected in case it

fails to meet any of the specified requirements. An agent of the contractor may be present at the making of the tests or they may be repeated in his presence.

15. In case of the failure of any of the tests, and if the contractor so desires, the engineer may, if he deem it to the interest of the United States, have any or all of the tests made or repeated by the Bureau of Standards, United States Department of Commerce and Labor, in the manner hereinafter specified, all expenses of such tests to be paid by the contractor. All such tests shall be made on samples furnished by the engineer.

STANDARD METHODS OF TESTING

Sampling.—16. The selection of the samples for testing will be left to the engineer. The number of packages sampled and the quantity to be taken from each package will depend on the importance of the work, the number of tests to be made, and the facilities for making them.

17. The samples should be so taken as to represent fairly the material, and, where conditions permit, at least 1 barrel in every 50 should be sampled. Before tests are made samples shall be passed through a sieve having 20 meshes per linear inch to remove foreign material. Samples shall be tested separately for physical qualities, but for chemical analysis mixed samples may be used. Every sample should be tested for soundness, but the number of tests for other qualities will be left to the discretion of the engineer.

Chemical Analysis.—18. The method to be followed for the analysis of cement shall be that proposed by the Committee on Uniformity in the Analysis of Materials for the Portland Cement Industry, reported in the Journal of the Society for Chemical Industry, volume 21, page 12, 1902, and published in Engineering News, volume 50, page 60, 1903, and in the Engineering Record, volume 48, page 49, 1903.

19. The insoluble residue shall be determined on a 1-gram sample, which is digested on the steam bath in hydrochloric acid of approximately 1.035 specific gravity until the cement is dissolved. The residue is filtered, washed with hot water, and the filter-paper contents digested on the steam bath in a 5 per cent solution of sodium carbonate. The residue is then filtered, washed with hot water, then with hot hydrochloric acid, approximately of 1.035 specific gravity, and finally with hot water, then ignited and weighed. The quantity so obtained is the insoluble residue.

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Determination of Specific Gravity.—20. The determination of specific gravity may be made with a standardized apparatus of Le Chatelier or other equally accurate form. Benzine (62° Baumé naphtha), or kerosene free from water should be used in making the determination. The cement should be allowed to pass slowly into the liquid of the volumenometer, taking care that the powder does not adhere to the sides of the graduated tube above the liquid and that the funnel through which it is introduced does not touch the liquid. The temperature of the liquid in the flask should not vary more than 1° F. during the operation. To this end the flask should be immersed in water. The results of repeated tests should agree within 0.01.

21. If the specific gravity of the cement as received is less than 3.10, a redetermination may be made as follows:

Seventy grams of the cement is placed in a nickel or platinum crucible about 2 inches in diameter and heated for 30 minutes at a temperature between 419° C. and 630° C. After the cement has cooled to atmospheric temperature the specific gravity shall be determined in the same manner as described above. The cement should be heated in a muffle or other suitable furnace, the temperature of which is to be maintained above the melting point of zinc (419° C.) but below the melting point of antimony (630° C.). This maximum temperature can be recognized as a very dull red which is just discernible in the dark.

Determination of Fineness.—22. The No. 100 and No. 200 sieves shall conform to the standard sieve specifications of the Bureau of Standards, Department of Commerce and Labor.

23. The determination of fineness should be made on a 50-gram sample, which may be dried at a temperature of 100° C. (212° F.) prior to sifting. The coarsely screened sample should be weighed and placed on the No. 200 sieve, which, with the pan and cover attached, should be held in one hand in a slightly inclined position and moved forward and backward in the plane of inclination, at the same time striking the side gently about 200 times per minute against the palm of the other hand on the upstroke. The operation is to be continued until not more than 0.05 gram will pass through in one minute. The residue should be weighed, then placed on the No. 100 sieve, and the operation repeated. The sieves should be thoroughly dry and clean. Determination of fineness may be made by washing the cement through the sieve or by a mechanical sifting device which has been previously standardized with the results obtained by hand sifting on equivalent samples. In case of the

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failure of the cement to pass the fineness requirements by the washing method or the mechanical device, it shall be tested by hand.

Mixing Cement Pastes and Mortars.—24. The quantity of cement or cement and sand to be used in the paste or mortar should be expressed in grams and the quantity of water in cubic centimeters. The material should be weighed, placed upon a non-absorbent surface, thoroughly mixed dry if sand be used, and a crater formed in the center, into which the proper percentage of clean water should be poured: the material on the outer edge should be turned into the crater by the aid of a trowel. As soon as the water has been absorbed the operation should be completed by vigorously mixing with the hands for one minute and a half. During the operation of mixing, the hands should be protected by rubber gloves. The temperature of the room and the mixing water should be maintained as nearly as practicable at 21° C. (70° F.).

Determination of Normal Consistency.—25. The normal consistency for neat paste to be used in making briquettes and pats should be determined by the ball method, as follows:

26. A quantity of cement paste should be mixed in the manner above described under Mixing Cement Pastes and Mortars, and quickly formed into a ball above 2 inches in diameter. The ball should then be dropped upon a hard, smooth, and flat surface from a height of 2 feet. The paste is of normal consistency when the ball does not crack and does not flatten more than one-half of its original diameter.

27. Trial pastes should be made with varying percentages of water until the correct consistency is obtained.

28. The percentage of water to be used in mixing mortars for sand briquettes is given by the formula:

$$y = \frac{2}{3} \frac{P}{n + 1} + K$$

in which y is the percentage of water required for the sand mortar, P is the percentage of water required for neat cement paste of normal consistency,

n is the number of parts of sand to one of cement by weight, and K is a constant which for standard Ottawa sand has the value 6.5.

The percentage of water to be used for mortars containing 3 parts standard Ottawa sand, by weight, to 1 of cement is indicated in the following statement:

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Percentage of water for neat cement paste	Percentage of water for 1 to 3 mortars of standard Ottawa sand
18.....	9.5
19.....	9.7
20.....	9.8
21.....	10.0
22.....	10.2
23.....	10.3
24.....	10.5
25.....	10.7
26.....	10.8
27.....	11.0
28.....	11.2
29.....	11.3

Determination of Soundness.—29. Pats of neat cement paste of normal consistency about 3 inches in diameter, one-half inch in thickness at the center, and tapering to a thin edge, should be kept in moist air for a period of 24 hours. One pat should then be kept in air and a second in water, at the ordinary temperature of the laboratory not to vary greatly from 21° C. (70° F.), and both observed at intervals for at least 28 days. A third pat should be exposed to steam at atmospheric pressure above boiling water for 5 hours.

Determination of Time of Setting.—30. The time of setting should be determined by the standardized Gillmore needles, as follows: A pat of neat cement paste about 3 inches in diameter and one-half inch in thickness with flat top, mixed at normal consistency, should be kept in moist air, at a temperature maintained as nearly as practicable at 21° C. (70° F.). The cement is considered to have acquired its initial set when the pat will bear, without appreciable indentation, a needle one-twelfth of an inch in diameter loaded to weigh one-fourth of a pound. The final set has been acquired when the pat will bear, without appreciable indentation, a needle one twenty-fourth of an inch in diameter, loaded to weigh 1 pound. In making the test the needle should be held in a vertical position and applied lightly to the surface of the pat. The pats made for the soundness test may be used to determine the time of setting.

Tensile Tests.—31. Tensile tests should be made on an approved machine. The test pieces shall be briquettes of the form recommended by the Committee on Uniform Tests of Cement of the American Society of Civil Engineers, and illustrated in Circular 33 of the Bureau of Standards. The briquettes shall be made of paste or mortar of normal consistency. Immediately after mixing, the paste or mortar should be placed in the molds, pressed in firmly by the fingers and smoothed off with a trowel without mechanical

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ramming. The material should be heaped above the mold, and in smoothing off, the trowel should be drawn over the mold in such a manner as to exert a moderate pressure on the material. The molds should be turned over and the operation of heaping and smoothing off repeated. Not less than three briquettes should be made and tested for each sample for each period of test. The neat tests are not considered so important as the sand tests. The briquettes should be broken as soon as they are removed from the water. The load should be applied at the rate of 600 pounds per minute.

Storage of Test Pieces.—32. During the first 24 hours after molding the test pieces should be kept in air sufficiently moist to prevent them from drying. After 24 hours in moist air the test pieces should be immersed in water. The air and water should be maintained as nearly as practicable at 21° C. (70° F.).

Standard Sand.—33. The sand to be used shall be natural sand from Ottawa, Ill., screened to pass a No. 20 sieve and retained on a No. 30 sieve.

34. Sand having passed the No. 20 sieve shall be considered standard when not more than 2 grams pass the No. 30 sieve after one minute continuous sifting of a 200-gram sample.

35. The No. 20 and No. 30 sieves shall conform to the standard sieve specifications of the Bureau of Standards, Department of Commerce and Labor.

II. METHODS OF CHEMICAL ANALYSIS

Prefatory Note.—While it may not be necessary to follow the standard method of analysis in routine tests when only a general indication of composition is desired, this method, including all precautions as stated in footnotes and italicized text, must always be followed when the results are to be used as the basis for rejection, or when an accurate knowledge of composition is desired.

The standard method can only yield accurate results in the hands of a careful and experienced analyst when all precautions are properly observed and even under these conditions the results obtained in the determinations of magnesia (MgO), sulphuric anhydride (SO₃), "loss on ignition" and "insoluble residue" may be ± 0.10 per cent in error, while in general results reported for magnesia tend to be too high. Under less favorable conditions the errors may be of much greater magnitude.

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It is desired to emphasize these points so as to prevent rejection of material if the specified limits are exceeded by less than 0.10 per cent.

CHEMICAL ANALYSIS

METHOD SUGGESTED FOR THE ANALYSIS OF LIMESTONES, RAW MIXTURES, AND PORTLAND CEMENT BY THE COMMITTEE ON UNIFORMITY IN TECHNICAL ANALYSIS WITH THE ADVICE OF W. F. HILLEBRAND

Report of Subcommittee (New York Section Society of Chemical Industry) on Uniformity in Analysis of Material for the Portland Cement Industry¹

(All matter printed in italics, both in text and footnotes, has been added during the preparation of this circular at the suggestion and with the approval of W. F. Hillebrand, with special application to the analysis of Portland Cement.)

Solution.—One-half gram² of the finely powdered substance is to be weighed out and, if a limestone or unburned mixture, strongly ignited in a covered platinum crucible over a strong blast for 15 minutes, or longer if the blast is not powerful enough to effect complete conversion to a cement in this time. It is then transferred to an evaporating dish, preferably of platinum for the sake of celerity in evaporation, moistened with enough water to prevent lumping, and 5 to 10 c.c. of strong HCl added and digested with the aid of gentle heat and agitation until solution is completed. Solution may be aided by light pressure with the flattened end of a glass rod.³ The solution is then evaporated to dryness, as far as this may be possible on the bath.

Silica (SiO₂).—The residue without further heating is treated at first with 5 to 10 c.c. of strong HCl, which is then diluted to half strength or less, or upon the residue may be poured at once a larger volume of acid of half strength. The dish is then covered and digestion allowed to go on for 10 minutes on the bath, after which the solution is filtered and the separated silica washed thoroughly with water. The filtrate is again evaporated to dryness, the residue without further heating taken up with acid and water, and the small amount of silica it contains separated on another filter paper. The papers containing the residue are transferred wet to a weighed platinum crucible, dried, ignited, first over a Bunsen burner until

¹ The original method was reported in the Journal of the Society for Chemical Industry, vol. 21, p. 30, but the method was subsequently modified by the committee, and the above text practically conforms to that in the Engineering Record, vol. 48, p. 49; Engineering News, vol. 50, p. 60.

² If a limestone, 0.75 gram should be used, the approximate equivalent of 0.5 gram of cement.

³ If anything remains undecomposed it should be separated, fused with a little Na₂CO₃, dissolved and added to the original solution. Of course a small amount of separated non-gelatinous silica is not to be mistaken for undecomposed matter.

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the carbon of the filter is completely consumed, and finally over the blast for 15 minutes and checked by a further blasting for 10 minutes or to constant weight.

The silica, if great accuracy is desired, is treated in the crucible with about 10 c.c. of HF and 4 drops⁴ H₂SO₄ and evaporated over a low flame to complete dryness. The small residue is finally blasted for a minute or two, cooled, and weighed. The difference between this weight and the weight previously obtained gives the amount of silica.⁵

Alumina and Iron (Al₂O₃ and FeO₃).—The filtrate, about 250 c.c. from the second evaporation for SiO₂, is made alkaline with NH₄OH after adding HCl, if need be, to insure a total of 10 to 15 c.c. strong acid,⁶ and boiled to expel excess of NH₃, or until there is but a faint odor of it, and the precipitated iron and aluminum hydroxides, after settling, are washed once by decantation and slightly on the filter. Setting aside the filtrate, the precipitate is dissolved in hot dilute HCl, the solution passing into the beaker in which the precipitation was made. The aluminum and iron are then reprecipitated by NH₄OH₆, boiled, and the second precipitate collected and washed on the filter used in the first instance. The filter paper with the precipitate is then placed in a weighed platinum crucible (*the one containing the residue from the silica if this was corrected by hydrofluoric acid treatment*), the paper burned off, and the precipitate, ignited and finally blasted 5 minutes, with care to prevent reduction, cooled and weighed as Al₂O₃+Fe₂O₃.⁷

Iron (Fe₂O₃).—The combined iron and aluminum oxides are fused in a platinum crucible at a very low temperature with about 3 to 4 grams KHSO₄, or, better, NaHSO₄,⁸ the melt taken up with so much dilute H₂SO₄ that there shall be no less than 5 grams absolute acid and enough water to effect solution on heating. The solution is then evaporated and eventually heated till acid fumes come off copiously. After cooling and redissolving in water the

⁴ 5 c.c. HF and 2 drops H₂SO₄ are sufficient.

⁵ For ordinary control in the plant laboratory this correction may, perhaps, be neglected; the double evaporation never. *The silica so found does not represent quite all in the material under analysis; a little has passed into the filtrate. Account should be taken of a possible loss in weight of the crucible itself, if the blast is very powerful.*

⁶ And 2 or 3 c.c. of bromine water. Bromine water is used for the purpose of collecting practically all the manganese here, instead of allowing it to distribute among several different precipitates.

⁷ This precipitate contains TiO₂, P₂O₅, Mn₃O₄.

⁸ Or the corresponding pyrosulphates which are less troublesome and more effective than the acid sulphates.

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small amount of silica is filtered out, weighed and corrected by HF and H_2SO_4 .⁹ The filtrate is reduced by zinc, or preferably by hydrogen sulphide, boiling out the excess of the latter afterwards while passing CO_2 through the flask, and titrated with permanganate.¹⁰

The strength of the permanganate solution should not be greater than 0.0040 g Fe_2O_3 per c.c.

Lime (CaO).—To the combined filtrate from the $\text{Al}_2\text{O}_3 + \text{Fe}_2\text{O}_3$ precipitate a few drops of NH_4OH are added, and the solution brought to boiling. To the boiling solution 20 c.c. of a saturated solution of ammonium oxalate is added, and the boiling continued until the precipitated CaC_2O_4 assumes a well-defined granular form. It is then allowed to stand for 20 minutes, or until the precipitate has settled, and then filtered and washed. The precipitate and filter are placed wet in a platinum crucible, and the paper is burned off over a small flame of a Bunsen burner. It is then ignited, re-dissolved in HCl , and the solution is made up to 100 c.c. with water. Ammonia is added in slight excess, and the liquid is boiled. If a small amount of Al_2O_3 separates this is filtered out, weighed, and the amount added to that found in the first determination, when greater accuracy is desired. The lime is then reprecipitated by ammonium oxalate, allowed to stand until settled, filtered and washed,¹¹ weighed as oxide after ignition and blasted in a covered crucible to constant weight, or determined with dilute standard permanganate.¹²

Magnesia (MgO).—The combined filtrates from the calcium precipitates are acidified with HCl and concentrated on the steam bath to about 150 c.c., 10 c.c. of saturated solution of $\text{Na}(\text{NH}_4)\text{-HPO}_4$ is added, and the solution boiled for several minutes. It is then removed from the flame and cooled by placing the beaker in ice water. After cooling, NH_4OH is added, drop by drop, with constant stirring until the crystalline ammonium-magnesium orthophosphate begins to form, and then in moderate excess, the stirring being continued for several minutes. It is then set aside

⁹ This correction of Al_2O_3 , Fe_2O_3 for silica should not be made when the HF correction of the main silica has been omitted, unless that silica was obtained by only one evaporation and filtration. After two evaporations and filtrations 1 to 2 mg. of SiO_2 are still to be found with the Al_2O_3 , Fe_2O_3 .

¹⁰ In this way only is the influence of titanium to be avoided and a correct result obtained for iron.

¹¹ The volume of wash water should not be too large; vide Hillebrand, *United States Geological Survey, Bull. 422, p. 110.*

¹² The accuracy of this method admits of criticism, but its convenience and rapidity demand its insertion.

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for several hours in a cool atmosphere and filtered. The precipitate is redissolved in hot dilute HCl, the solution made up to about 100 c.c., 1 c.c. of a saturated solution of Na (NH₄)HPO₄ added, and ammonia, drop by drop, with constant stirring until the precipitate is again formed as described and the ammonia is in moderate excess. It is then allowed to stand for about 2 hours,¹³ when it is filtered on a paper or a Gooch crucible, ignited, cooled, and weighed as Mg₂P₂O₇.

The pyrophosphate invariably contains calcium which can be determined as follows:

Dissolve the ignited pyrophosphate in a little dilute H₂SO₄ and add enough absolute alcohol to make about 90 to 95 per cent of the final volume. After several hours collect the small and sometimes almost invisible precipitate of calcium sulphate on a small filter and wash it free of phosphoric acid with alcohol. Dry the filter and extract from it the precipitate by a few cubic centimeters of hot water acidulated with HCl. Make this solution alkaline with ammonia, throw in a few crystals of ammonium oxalate and continue heating till a precipitate becomes visible. After an hour filter, wash and ignite to calcium oxide. Its weight, averaging perhaps 0.5 mg., is to be added to that of the lime already found and subtracted as tricalcium phosphate (not pyrophosphate) from that of the magnesium pyrophosphate.

In order to determine approximately the iron and aluminum present the following procedure may be followed:

Evaporate the alcoholic filtrate from the calcium sulphate and heat the residue to destroy separated organic matter. Take the residue up with a little HCl and water and when dissolved add a drop of bromine water. Add ammonia till the magnesia is again precipitated and let stand for an hour. Decant most of the supernatant liquid and add slowly, drop by drop, acetic acid till all fine-grained matter has dissolved. Usually there will remain a little flocculent matter which is likely to consist in greater part or wholly of phosphates of iron and aluminum (and manganese if this last was not removed by bromine and ammonia as in the section on Alumina and Iron Oxides). After ignition the precipitate often shows a reddish color. Unless great care is exercised this separation will lead to erroneous results, either by inclusion of magnesium with the impurities as weighed or by loss of these in consequence of using too much acetic acid.

Alkalies (K₂O and Na₂O).—For the determination of the alkalies, the well-known method of Prof. J. Lawrence Smith is to

¹³ A paper filter should always be used if the pyrophosphate is to be corrected for contaminations.

be followed, either with or without the addition of CaCO_2 with NH_4Cl .

Sulphuric Anhydride Acid (SO_3).—One gram of the substance is dissolved in 15 c.c. (5 c.c.) of HCl , and 45 c.c. water, filtered, and the residue washed thoroughly.¹⁴

The solution is made up to 250 c.c. in a beaker and boiled. To the boiling solution 10 c.c. of a saturated solution of BaCl_2 ¹⁵ is added slowly, drop by drop, from a pipette and the boiling continued until the precipitate is well formed, or digested on the steam bath may be substituted for the boiling. It is then set aside overnight, or for a few hours, filtered, ignited, and weighed as BaSO_4 .

Total Sulphur.—One gram of the material is weighed out in a large platinum crucible and fused with Na_2CO_3 and a little KNO_3 , being careful to avoid contamination from sulphur in the gases from source of heat. This may be done by fitting the crucible in a hole in an asbestos board.

The melt is treated in the crucible with boiling water and the liquid poured into a tall narrow beaker and more hot water added until the mass is disintegrated. The solution is then filtered. The filtrate contained in a No. 4 beaker is to be acidulated with HCl and made up to 250 c.c. with distilled water, boiled, the sulphur precipitated as BaSO_4 and allowed to stand overnight, or for a few hours.

The following procedure is in accordance with the recommendation of W. F. Hillebrand in United States Geological Survey, Bulletin 422, page 227:

In a platinum crucible mix 1 gram of the sample with one-half gram of sulphur-free sodium carbonate. Place the covered crucible in a hole in an asbestos board that is held in a somewhat inclined position and apply a blast flame upon the crucible below the asbestos for 10 to 15 minutes. Transfer the sintered mass to a beaker and cover with water. Cleanse the crucible with dilute hydrochloric acid and pour the solution into the beaker. Add more acid till decomposition is complete in the cold or on gently warming. Filter, wash with hot water, dilute to 150 to 200 c.c., boil, and precipitate with barium chloride.

It should be borne in mind that by neither of the methods given is a barium sulphate obtained that is perfectly pure. Ferric (and if present alkali) sulphate, also barium chloride, contaminate it and it is im-

¹⁴ Evaporation to dryness is unnecessary unless gelatinous silica should have separated, and should never be performed on a bath heated by gas; vide Hillebrand: *United States Geological Survey, Bulletin 422, p. 198.*

¹⁵ Ten per cent. solution is preferable to one that is saturated.

GOVERNMENT SPECIFICATION FOR PORTLAND CEMENT

possible to correct for them directly. The most convenient way to obtain a correction is by a blank with a solution containing sulphur and the other main constituents of the cement in approximately the amounts and proportions found in the test sample.

Loss on Ignition.—Half a gram of cement is to be weighed out in a (*covered*) platinum crucible, placed in a hole in an asbestos board so that about three-fifths of the crucible projects below, and blasted 15 minutes, preferably with an inclined flame. The loss by weight, which is checked by a second blasting of 5 minutes, is the loss on ignition.

Recent investigations have shown that large errors in results are often due to the use of impure distilled water and reagents. The analyst should, therefore, test his distilled water by evaporation and his reagents by appropriate tests before proceeding with his work.

Insoluble Residue.—*The insoluble residue¹⁶ shall be determined on a 1-gram sample which is digested on the steam bath in hydrochloric acid of approximately 1.035 specific gravity until the cement is dissolved. The residue is filtered, washed with hot water, and the filter paper contents digested on the steam bath in a 5 per cent solution of sodium carbonate. The residue is then filtered, washed with hot water, then with hot hydrochloric acid, approximately of 1.035 specific gravity, and finally with hot water, then ignited and weighed. The quantity so obtained is the insoluble residue.*

III. INTERPRETATION OF RESULTS

CHEMICAL

The composition of normal Portland cement has been the subject of a great deal of investigation and it can be said that the quantities of silica, alumina, oxide of iron, lime, magnesia, and sulphuric anhydride can vary within fairly wide limits without materially affecting the quality of the material.

A normal American Portland cement which meets the standard specifications for soundness, setting time and tensile strength has an approximate composition within the following limits:

¹⁶ This determination was not considered by the Committee of the Society of Chemical Industry, and is reproduced from paragraph 19 of the United States Government specification for Portland cement.

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	Per Cent
Silica.....	19-25
Alumina.....	5-9
Iron oxide.....	2-4
Lime.....	60-64
Magnesia.....	1-4
Sulphur trioxide.....	1-1.75
Loss on ignition.....	0.5-3.00
Insoluble residue.....	0.1-1.00

It is also true that a number of cements have been made both here and abroad which have passed all standard physical tests in which these limits have been exceeded in one or more particulars, and it is equally true that a sound and satisfactory cement does not necessarily result from the above composition.

It is probable that further investigation will give a clearer understanding of the constitution of Portland cement, but at present chemical analysis furnishes but little indication of the quality of the material.

Defective cement usually results from imperfect manufacture, not from faulty composition. Cement made from very finely ground material, thoroughly mixed and properly burned, may be perfectly sound when containing more than the usual quantity of lime, while a cement low in lime may be entirely unsound due to careless manufacture.

The analysis of a cement will show the uniformity in composition of the product from individual mills, but will furnish little or no indication of the quality of the material. Occasional analysis should, however, be made for record and to determine the quantity of sulphuric anhydride and magnesia present.

The ground clinker as it comes from the mill is usually quick setting which requires correction. This is usually accomplished by the addition of a small quantity of more or less hydrated calcium sulphate, either gypsum or plaster of Paris. Experience and practice have shown that an addition of 3 per cent or less is sufficient for the purpose.

Three per cent of calcium sulphate (CaSO_4) contains about 1.75 per cent sulphuric anhydride (SO_3), and as this has been considered the maximum quantity necessary to control time of set, the specification limits the SO_3 content to 1.75 per cent.

The specification prohibits the addition of any material subsequent to calcination except the 3 per cent of calcium sulphate permitted to regulate time of set. Other additions may be difficult or impossible to detect even by a careful mill inspection during the process of manufacture, but as the normal adulterant would be

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ground raw material, an excess of "insoluble residue" would reveal the addition of silicious material, and an excess in "loss on ignition" would point to the addition of calcareous material when either is added in sufficient quantity to make the adulteration profitable.

The effect of relatively small quantities of magnesia (MgO) in normal Portland cement, while still under investigation, can be considered harmless. Earlier investigators believed that as magnesia had a slower rate of hydration than lime, the hydration of any free magnesia (MgO) present would occur after the cement had set and cause disintegration.

The effect of magnesia was considered especially injurious when the cement was exposed to the action of sea water. More recent investigation has shown that cement can be made which is perfectly sound under all conditions when containing 5 per cent of magnesia and it has also been found that the lime in Portland cement exposed to sea water is replaced by magnesia.

The maximum limit for magnesia has been set at 4 per cent, as it has been established that this quantity is not injurious and it is high enough to permit the use of the large quantities of raw material available in most sections of the country.

PHYSICAL

Specific Gravity.—If the Le Chatelier apparatus is used for the determination of specific gravity, the clean volumometer flask is filled with benzine free from water (which can be obtained by placing some calcium chloride or caustic lime in the benzine storage jar) to a point on the stem between zero and 1 cubic centimeter. The flask is then placed in a constant temperature bath until volume is constant. The usual method is to introduce 64 grams of cement into the flask, taking care that the powder does not adhere to the tube above the liquid, and to free the cement from air by rolling the flask in an inclined position. The flask is then replaced in the constant temperature bath until a constant volume is recorded.

The specific gravity is obtained from the formula:

$$\text{specific gravity} = \frac{\text{weight of cement in grams}}{\text{displaced volume in cu. cm.}}$$

The specific gravity of a Portland cement is not an indication of its cementing value. It will vary with the constituents of the cement, especially with the content of iron oxide. Thus the white or very light Portland cements, containing only a fraction of a per

cent. of iron oxide, usually have a comparatively low specific gravity ranging from 3.05 to 3.15, while a cement containing 3 to 4 per cent. or more of iron oxide may have a specific gravity of 3.20 or even higher. It is materially affected by the temperature and duration of burning the cement, the hard-burned cement having the higher specific gravity. A comparatively low specific gravity does not necessarily indicate that a cement is underburned or adulterated, as large percentages of raw materials could be added to a cement with a normally high specific gravity before the gravity would be reduced below 3.10.

If a Portland cement fresh from the mill normally has a comparatively low specific gravity, upon aging it may absorb sufficient moisture and carbon dioxide to reduce the gravity below 3.10. It has been found that this does not appreciably affect the cementing value of the material; in fact, many cements are unsound until they have been aged. Thus a redetermination is permitted upon a sample heated to a temperature sufficient to drive off any moisture which may be absorbed by the cement subsequent to manufacturing, but would not drive off any carbon dioxide nor correct underburning in the process of manufacturing the cement.

The value of the specific gravity determination lies in the fact that it is easily made in the field or laboratory, and when the normal specific gravity of the cement is known, any considerable variation in quality due to underburning or the addition of foreign materials may be detected.

Fineness.—Only the extremely fine powder of cement called flour possesses appreciable cementing qualities and the coarser particles are practically inert. No sieve is fine enough to determine the flour in a cement, nor is there any other means of accurately and practically measuring the flour. Some cements grind easier than others, thus, although a larger percentage of one cement may pass the 200-mesh sieve than another, the former may have a smaller percentage of actual flour due to the difference in the hardness and the character of the clinker, and the method used in grinding. Thus the cementing value of different cements can not be compared directly upon their apparent fineness through a 200-mesh sieve. With cement from the same mill, with similar clinker and grinding machinery, however, it is probable that the greater the percentage which passes the 200-mesh sieve the greater the percentage of flour in that particular cement.

Normal Consistency.—The quantity of water used in making the paste from which the pats for soundness, tests of setting, and

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the briquettes are made, is very important and may vitally affect the results obtained. The determination consists in measuring the quantity of water required to bring a cement to a certain state of plasticity.

In determining the normal consistency by the ball method, after mixing the paste it should be formed into a ball with as little working as possible and a new batch of cement should be mixed for each trial paste. In order to obtain just the requisite quantity of paste

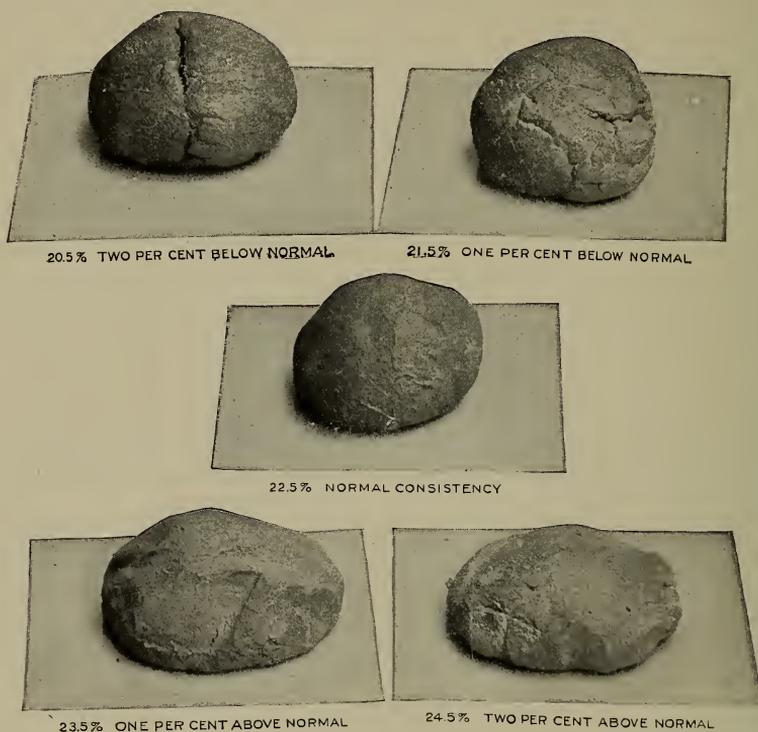


Fig. 1.—Appearance of Ball for Different Consistencies of Cement Paste

to form a ball 2 inches in diameter, a measure made from a pipe with a 2-inch inside diameter cut $1\frac{1}{3}$ inches long will be found convenient. The section of pipe should be open at both ends, so that it can be pushed down onto the paste on the mixing table and the excess paste cut off with the trowel. The appearance of the ball using the correct percentage of water for normal consistency as compared with a less and greater quantity of water is shown in Fig. 1.

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Mixing.—The homogeneity of the cement paste is dependent upon the thoroughness of the mixing, and this may have considerable influence upon the time of setting and the strength of the briquettes.

Soundness.—The purpose of this test is to detect those qualities in a cement which tend to destroy the strength and durability. Unsoundness is usually manifested by a change in volume which causes cracking, swelling, or disintegration. If the pat is not properly made, or if it is placed where it will be subject to any drying during the first 24 hours, it may develop what are known as shrinkage cracks, which are not an indication of unsoundness and should not be confused with disintegration cracks, as shown in Figs. 2 and 3. No shrinkage cracks should develop after the first 24 or 48 hours.

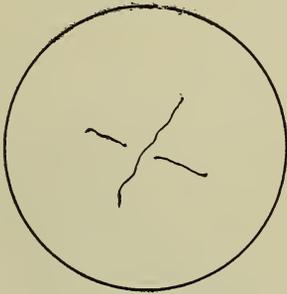


Fig. 2.—Soundness Pat Showing Shrinkage Cracks

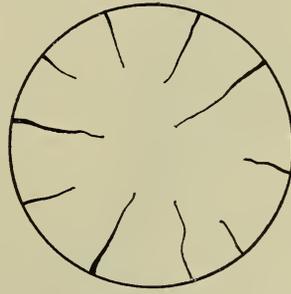


Fig. 3.—Soundness Pat Showing Disintegration Cracks

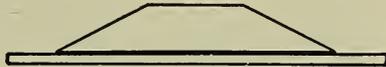


Fig. 5.—Soundness Pat with Top Surface Flattened for Determining Time of Setting

The failure of the pats to remain on the glass nor the cracking of the glass to which the pat is attached does not necessarily indicate unsoundness. In molding the pats, the cement paste should first be flattened on the glass and the pat formed by drawing the trowel from the outer edge toward the center, as shown in Fig. 4.

Time of Setting.—The purpose of this test is to determine the time which elapses from the moment water is added until the paste ceases to be plastic and the time required for it to obtain a certain degree of hardness. The determination of the “initial set” or when plasticity ceases is the more important, as a disturbance of the material after this time may cause a loss of strength and thus it is important that the mixing and molding or the incorporating

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of the material into the work be accomplished within this time. The time of setting is usually determined upon one of the pats which is to be used for the soundness test, the top surface being flattened somewhat, as shown in Fig. 5. In using the Gillmore needles care should be taken to apply the needles in a vertical position and perpendicular to the surface of the pat. Fig. 6 shows an arrangement for mounting the Gillmore needles so that they are always perpendicular to the surface of the pat. The rate of setting and hardening may be materially affected by slight changes in temperature. The percentage of water used in gauging and the hu-

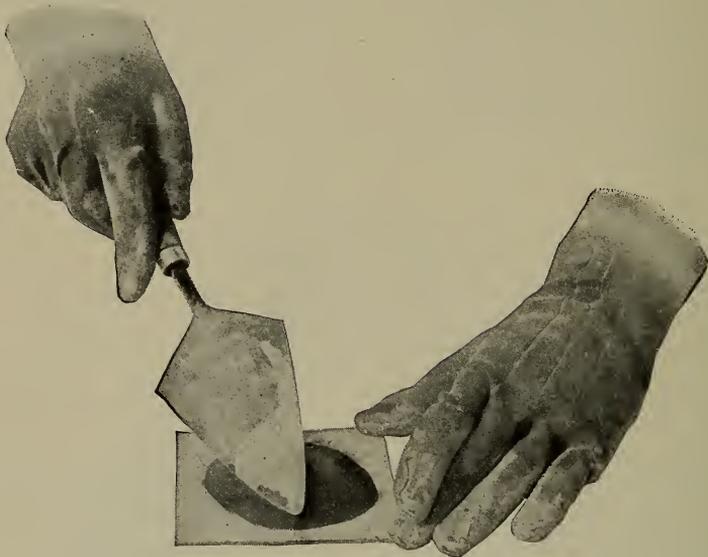


Fig. 4.—Correct Method of Molding Cement Pat

midity of the moist closet in which the test pieces are stored may also affect the setting somewhat.

Tensile Tests.—Consistent results can only be obtained by exercising great care in molding and testing the briquettes. The correct method of filling the mold is shown in Figs. 7 and 8. In testing, the sides of the briquettes and the clips should be thoroughly cleaned and free from grains of sand or dirt which would prevent a good bearing, and the briquette should be carefully centered in the clips so as to avoid cross strains. It may be considered good laboratory practice if the individual briquettes of any set do not show a greater variation from the mean value than 8 per cent for sand mixtures and 12 per cent for neat mixtures.

IV. AUXILIARY SPECIFICATIONS

BUREAU OF STANDARDS SIEVE SPECIFICATIONS

Wire cloth for standard sieves for cement and sand shall be woven (not twilled) from brass, bronze, or other suitable wire, and mounted on the frames without distortion.

The sieve frames shall be circular, about 20 centimeters (7.87 inches) in diameter, 6 centimeters (2.36 inches) high, and provided with a pan about 5 centimeters (1.97 inches) deep and a cover.

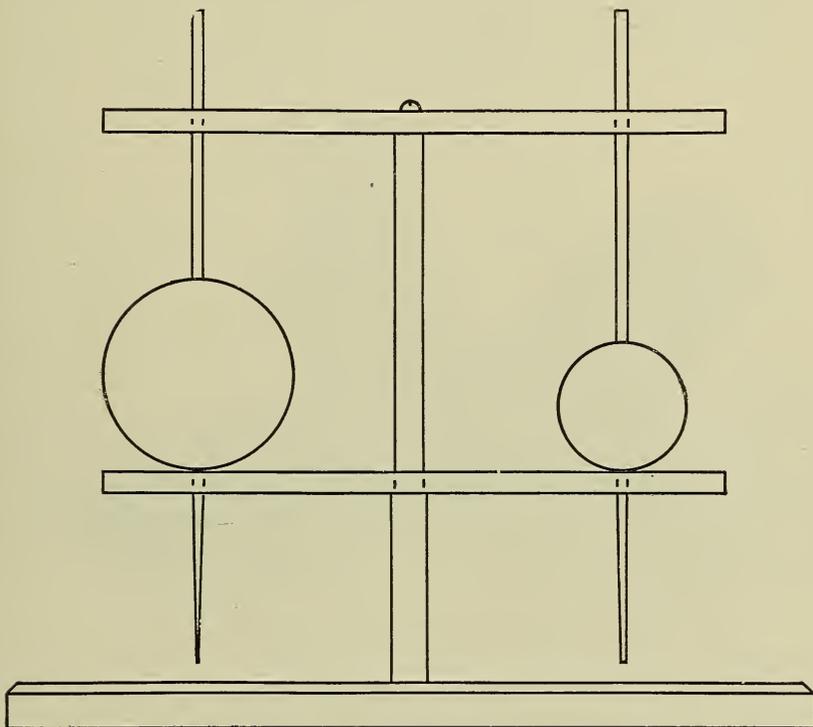


Fig. 6.—Method of Mounting Gillmore Needles

No. 100 Cement Sieve, 0.0055-Inch Opening.—The No. 100 sieve should have 100 wires per inch and shall conform to the following specifications of diameter of wire and size of mesh:

The diameter of the wires in the sieve should be 0.0045 inch and the average diameter of such wires as may be measured shall not be outside the limits 0.0042 to 0.0048 inch for either warp or shoot wires. The number of warp wires per whole inch, as measured at any point of the sieve, shall not be outside the limits 98 to 101

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per inch, and of the shoot wires 96 to 102 per inch. For any interval of 0.25 to 0.50 inch in which the mesh may be measured the mesh shall not be outside the limits 95 to 101 wires per inch for the warp wires and 93 to 103 wires per inch for the shoot wires.

No. 200 Cement Sieve, 0.0029-Inch Opening.—The No. 200 sieve should have 200 wires per inch and shall conform to the following specifications of diameter of wire and size of mesh:

The diameter of the wires in the sieve should be 0.0021 inch, and the average diameter of such wires as may be measured shall not be outside the limits 0.0019 to 0.0023 inch for either warp or shoot wires. The number of warp wires per whole inch, as measured at any point of the sieve, shall not be outside the limits 195 to 202

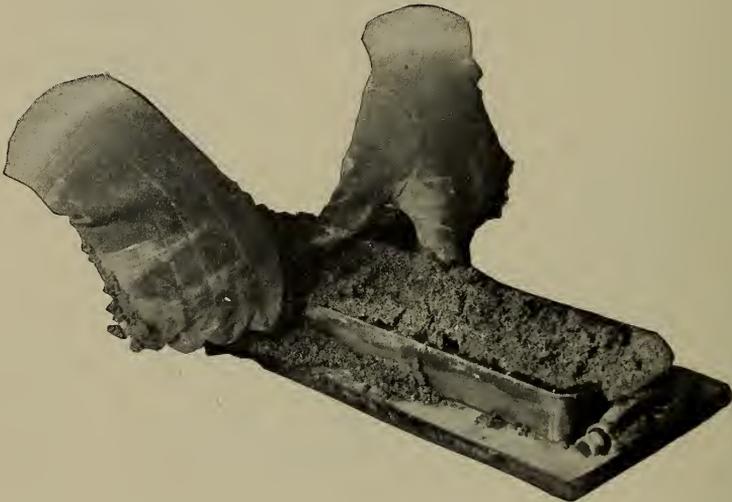


Fig. 7.—Correct Method of Filling Briquette Mold

per inch, and of the shoot wires 192 to 204 per inch. For any interval of 0.25 to 0.50 inch in which the mesh may be measured the mesh shall not be outside the limits 192 to 203 wires per inch for the warp wires and 190 to 205 wires per inch for the shoot wires.

No. 20 Sand Sieve, 0.0335-Inch Opening.—No. 20 sieves shall have between 19.5 and 20.5 wires per whole inch of the warp wires and between 19 and 21 wires per inch of the shoot wires. The diameter of the wire should be 0.0165 inch and the average as measured shall not vary outside the limits 0.0160 to 0.0170 inch.

No. 30 Sand Sieve, 0.0223-Inch Opening.—No. 30 sieves shall have between 29.5 and 30.5 wires per whole inch of the warp wires

GOVERNMENT SPECIFICATION FOR PORTLAND CEMENT

and between 28.5 and 31.5 per whole inch of the shoot wires. The diameter of the wire should be 0.0110 inch and the average as measured shall not vary outside the limits 0.0105 to 0.0115 inch.

BUREAU OF STANDARDS SPECIFICATION FOR SPECIFIC-GRAVITY FLASKS

Material and Annealing.—The material from which the flasks are made shall be glass of the best quality, transparent, and free from striæ. It shall adequately resist chemical action and have small thermal hysteresis. The flasks shall be thoroughly annealed at 400° C. to 500° C. for 24 hours and allowed to cool slowly before

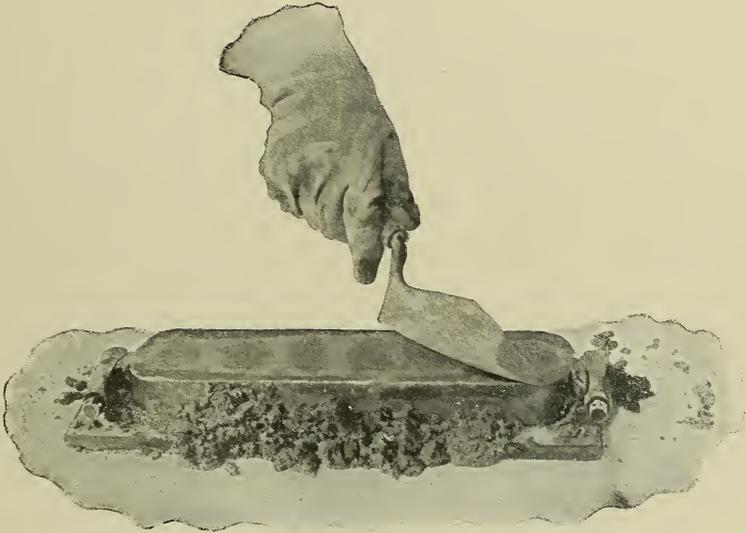


Fig. 8.—Correct Method of Troweling Surface of Briquettes

being graduated. They shall be of sufficient thickness to insure reasonable resistance to breakage.

Design.—The cross-section of the flask shall be circular and the shape and dimensions shall conform to the diagram shown in Fig. 9. This design is intended to insure complete drainage of the flask on emptying and stability of standing on a level surface, as well as accuracy and precision of reading. The neck of the flask shall be cylindrical for at least 1 centimeter above and below every graduation mark. There shall be a space of at least 1 centimeter between the highest graduation mark and the lowest point of the grinding for the glass stopper.

GOVERNMENT SPECIFICATION FOR PORTLAND CEMENT

the 1-c.c. graduation. The graduations shall be of uniform width, finely but distinctly etched, and shall be perpendicular to the axis of the flask. The 0.1-c.c. graduations shall be at least 1 millimeter apart. This will require an internal diameter of the neck not greater than 11.3 millimeters. The 1-c.c. graduations shall extend completely around the neck of the flask and shall be numbered to indicate the capacity. The 0.1-c.c. graduations shall extend at least halfway around the neck and the 0.5-c.c. graduations shall have a length about midway between the other two. The graduation marks shall have no apparent irregularities of spacing.

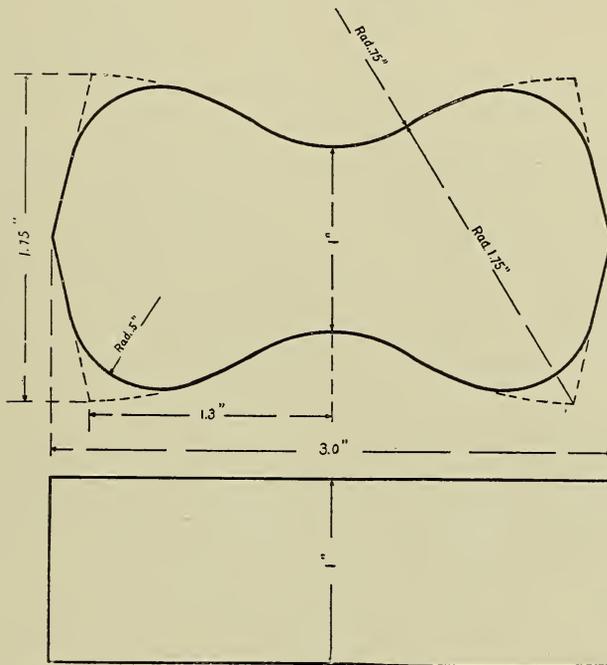


Fig. 10.—Form of Briquette as Recommended by the Committee on Uniform Tests of Cement of the American Society of Civil Engineers

Standard Temperature.—The flasks shall be standard at 20° C. The indicated specific gravities will then be at 20° referred to water at 4° as unity—that is, density at 20° in grams per c.c.

Inscriptions.—Each flask shall bear a permanent identification number and the stopper shall bear the same number. The standard temperature shall be indicated and the unit of capacity shall be shown by the letters “c.c.” placed above the highest graduation mark.

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Tolerance.—The error of any indicated capacity shall not be greater than 0.05 c.c.

Interpretation of Specification.—The foregoing specification is intended to represent the most desirable form of specific gravity flask for use in testing cements. Variations of a few millimeters in such dimensions as total height of flask, diameter of base, etc., are to be expected and will not be considered sufficient cause for rejection. The specification in regard to tolerance, inscriptions, length, spacing, and uniformity of graduations will, however, be rigidly enforced.

STANDARD SPECIFICATIONS FOR CEMENT

As Adopted August 16, 1909, by the American Society for
Testing Materials

GENERAL OBSERVATIONS

1. These remarks have been prepared with a view of pointing out the pertinent features of the various requirements and the precautions to be observed in the interpretation of the results of the tests.

2. The Committee would suggest that the acceptance or rejection under these specifications be based on tests made by an experienced person having the proper means for making the tests.

Specific Gravity.—3. Specific gravity is useful in detecting adulteration. The results of tests of specific gravity are not necessarily conclusive as an indication of the quality of a cement, but when in combination with the results of other tests may afford valuable indications.

Fineness.—4. The sieves should be kept thoroughly dry.

Time of Setting.—5. Great care should be exercised to maintain the test pieces under as uniform conditions as possible. A sudden change or wide range of temperature in the room in which the tests are made, a very dry or humid atmosphere, and other irregularities vitally affect the rate of setting.

Constancy of Volume.—6. The tests for constancy of volume are divided into two classes, the first normal, the second accelerated. The latter should be regarded as a precautionary test only, and not infallible. So many conditions enter into the making and interpreting of it that it should be used with extreme care.

7. In making the pats the greatest care should be exercised to avoid initial strains due to molding or to too rapid drying-out during the first twenty-four hours. The pats should be preserved under the most uniform conditions possible, and rapid changes of temperature should be avoided.

8. The failure to meet the requirements of the accelerated tests need not be sufficient cause for rejection. The cement may,

STANDARD SPECIFICATIONS FOR CEMENT

however, be held for 28 days, and a retest made at the end of that period using a new sample. Failure to meet the requirements at this time should be considered sufficient cause for rejection, although in the present state of our knowledge it cannot be said that such failure necessarily indicates unsoundness, nor can the cement be considered entirely satisfactory simply because it passes the tests.

SPECIFICATIONS

General Conditions.—1. All cement shall be inspected.

2. Cement may be inspected either at the place of manufacture or on the work.

3. In order to allow ample time for inspecting and testing, the cement should be stored in a suitable weather-tight building having the floor properly blocked or raised from the ground.

4. The cement shall be stored in such a manner as to permit easy access for proper inspection and identification of each shipment.

5. Every facility shall be provided by the contractor and a period of at least 12 days allowed for the inspection and necessary tests.

6. Cement shall be delivered in suitable packages with the brand and name of manufacturer plainly marked thereon.

7. A bag of cement shall contain 94 pounds of cement net. Each barrel of Portland cement shall contain 4 bags, and each barrel of natural cement shall contain 3 bags of the above net weight.

8. Cement failing to meet the 7-day requirements may be held awaiting the results of the 28-day tests before rejection.

9. All tests shall be made in accordance with the methods proposed by the Committee on Uniform Tests of Cement of the American Society of Civil Engineers, presented to the Society January 21, 1903, amended January 20, 1904, and January 15, 1908, with all subsequent amendments thereto.*

10. The acceptance or rejection shall be based on the following requirements:

Natural Cement.—11. *Definition.*—This term shall be applied to the finely pulverized product resulting from the calcination of an argillaceous limestone at a temperature only sufficient to drive off the carbonic acid gas.

Fineness.—12. It shall leave by weight a residue of not more

* See pp. 7-22.—Ed.

STANDARD SPECIFICATIONS FOR CEMENT

than 10 per cent on the No. 100, and 30 per cent on the No. 200 sieve.

Time of Setting.—13. It shall not develop initial set in less than 10 minutes, and shall not develop hard set in less than 30 minutes, or in more than 3 hours.

Tensile Strength.—14. The minimum requirements for tensile strength for briquettes 1 square inch in cross section shall be as follows, and the cement shall show no retrogression in strength within the periods specified:

<i>Age.</i>	<i>Neat Cement.</i>	<i>Strength.</i>
24 hours in moist air		75 lbs.
7 days (1 day in moist air, 6 days in water)		150 "
28 days (1 day in moist air, 27 days in water)		250 "
<i>One Part Cement, Three Parts Standard Ottawa Sand.</i>		
7 days (a day in moist air, 6 days in water)		50 "
28 days (1 day in moist air, 27 days in water)		125 "

Constancy of Volume.—15. Pats of neat cement about 3 inches in diameter, one-half inch thick at center, tapering to a thin edge, shall be kept in moist air for a period of 24 hours.

(a) A pat is then kept in air at normal temperature.

(b) Another is kept in water maintained as near 70° F. as practicable.

16. These pats are observed at intervals for at least 28 days, and, to satisfactorily pass the tests shall remain firm and hard and show no signs of distortion, checking, cracking or disintegrating.

Portland Cement.—17. *Definition.*—This term is applied to the finely pulverized product resulting from the calcination to incipient fusion of an intimate mixture of properly proportioned argillaceous and calcareous materials, and to which no addition greater than 3 per cent has been made subsequent to calcination.

Specific Gravity.—18. The specific gravity of cement shall be not less than 3.10. Should the test of cement as received fall below this requirement, a second test may be made upon a sample ignited at low red heat. The loss in weight of the ignited cement shall not exceed 4 per cent.

Fineness.—19. It shall leave by weight a residue of not more than 8 per cent on the No. 100, and not more than 25 per cent on the No. 200 sieve.

Time of Setting.—20. It shall not develop initial set in less than 30 minutes, and must develop hard set in not less than 1 hour, nor more than 10 hours.

STANDARD SPECIFICATIONS FOR CEMENT

Tensile Strength.—21. The minimum requirements for tensile strength for briquettes 1 square inch in cross section shall be as follows, and the cement shall show no retrogression in strength within the periods specified:

<i>Age.</i>	<i>Neat Cement.</i>	<i>Strength.</i>
24 hours in moist air.....		175 lbs.
7 days (1 day in moist air, 6 days in water).....		500 “
28 days (1 day in moist air, 27 days in water).....		600 “
<i>One Part Cement, Three Parts Standard Ottawa Sand.</i>		
7 days (1 day in moist air, 6 days in water).....		200 “
28 days (1 day in moist air, 27 days in water).....		275 “

Constancy of Volume.—22. Pats of neat cement about 3 inches in diameter, one-half inch thick at the center, and tapering to a thin edge shall be kept in moist air for a period of 24 hours.

(a) A pat is then kept in air at normal temperature and observed at intervals for at least 28 days.

(b) Another pat is kept in water maintained as near 70° F. as practicable, and observed at intervals for at least 28 days.

(c) A third pat is exposed in any convenient way in an atmosphere of steam, above boiling water, in a loosely closed vessel for 5 hours.

23. These pats, to satisfactorily pass the requirements, shall remain firm and hard and show no signs of distortion, checking, cracking or disintegrating.

Sulphuric Acid and Magnesia.—24. The cement shall not contain more than 1.75 per cent of anhydrous sulphuric acid (SO₃), nor more than 4 per cent of magnesia (MgO).

METHODS FOR TESTING CEMENT*

REPORT OF THE COMMITTEE OF THE AMERICAN SOCIETY OF
CIVIL ENGINEERS, PRESENTED AT THE ANNUAL
MEETING, JANUARY 17, 1912

SAMPLING

1. **Selection of Sample.**—The selection of samples for testing should be left to the engineer. The number of packages sampled and the quantity taken from each package will depend on the importance of the work and the facilities for making the tests.

2. The samples should fairly represent the material. When the amount to be tested is small it is recommended that 1 barrel in 10 be sampled; when the amount is large it may be impracticable to take samples from more than 1 barrel in 30 or 50. When the samples are taken from bins at the mill 1 for each 50 to 200 barrels will suffice.

3. Samples should be passed through a sieve having 20 meshes per linear inch, in order to break up lumps and remove foreign material; the use of this sieve is also effective to obtain a thorough mixing of the samples when this is desired. To determine the acceptance or rejection of cement it is preferable, when time permits, to test the samples separately. Tests to determine the general characteristics of a cement, extending over a long period, may be made with mixed samples.

4. **Method of Sampling.**—Cement in barrels should be sampled through a hole made in the head, or in one of the staves midway between the heads, by means of an auger or a sampling iron similar to that used by sugar inspectors; if in bags, the sample should be taken from surface to center; cement in bins should be sampled in such a manner as to represent fairly the contents of the bin. Sampling from bins is not recommended if the method of manufacture is such that ingredients of any kind are added to the cement subsequently.

CHEMICAL ANALYSIS

5. **Significance.**—Chemical analysis may serve to detect adul-

* Accompanying Final Report of Special Committee of American Society of Civil Engineers on Uniform Tests of Cement, dated January 17th, 1912.

teration of cement with inert material, such as slag or ground limestone, if in considerable amount. It is useful in determining whether certain constituents, such as magnesia and sulphuric anhydride, are present in inadmissible proportions.

6. The determination of the principal constituents of cement, silica, alumina, iron oxide, and lime, is not conclusive as an indication of quality. Faulty cement results more frequently from imperfect preparation of the raw material or defective burning than from incorrect proportions. Cement made from material ground very finely and thoroughly burned may contain much more lime than the amount usually present, and still be perfectly sound. On the other hand, cements low in lime may, on account of careless preparation of the raw material, be of dangerous character. Furthermore, the composition of the product may be so greatly modified by the ash of the fuel used in burning as to affect in a great degree the significances of the results of analysis.

7. **Methods.**—The methods to be followed, except for determining the loss on ignition, should be those proposed by the Committee on Uniformity in the Analysis of Materials for the Portland Cement Industry, reported in the *Journal* of the Society for Chemical Industry, Vol. 21, page 12, 1902; and published in *Engineering News*, Vol. 50, page 60, 1903; and in *Engineering Record*, Vol. 48, page 49, 1903, and in addition thereto, the following:

(a) The insoluble residue may be determined as follows: To a 1-gram sample of cement are added 30 cu. cm. of water and 10 cu. cm. of concentrated hydrochloric acid, and then warmed until effervescence ceases, and digested on a steam bath until dissolved. The residue is filtered, washed with hot water, and the filter paper and contents digested on the steam bath in a 5% solution of sodium carbonate. This residue is filtered, washed with hot water, then with hot hydrochloric acid, and finally with hot water, and then ignited at a red heat and weighed. The quantity so obtained is the insoluble residue.

(b) The loss on ignition shall be determined in the following manner: $\frac{1}{2}$ gram of cement is heated in a weighed platinum crucible, with cover, for 5 minutes with a Bunsen burner (starting with a low flame and gradually increasing to its full height) and then heated for 15 minutes with a blast lamp: the difference between the weight after cooling and the original weight is the loss on ignition. The temperature should not exceed 900° cent. or a low red heat; the ignition should preferably be made in a muffle.

METHODS FOR TESTING CEMENT

SPECIFIC GRAVITY

8. **Significance.**—The specific gravity of cement is lowered by adulteration and hydration, but the adulteration must be considerable to be detected by tests of specific gravity.

9. Inasmuch as the differences in specific gravity are usually very small, great care must be exercised in making the determination.

10. **Apparatus.**—The determination of specific gravity should be

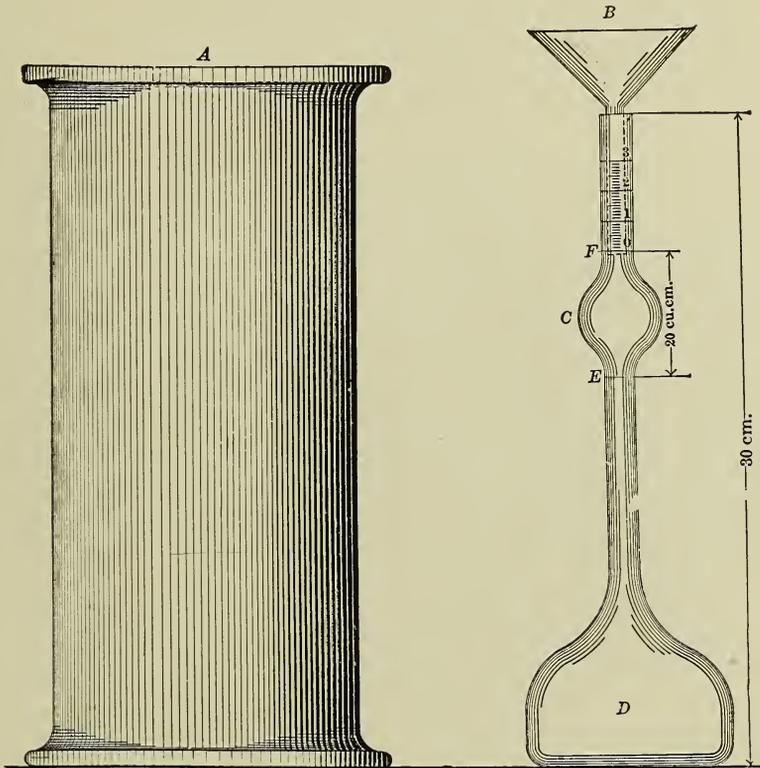


Fig. 1.—Le Chatelier's Specific-Gravity Apparatus

made with a standardized Le Chatelier apparatus. This consists of a flask (*D*), Fig. 1, of about 120 cu. cm. capacity, the neck of which is about 20 cm. long; in the middle of this neck is a bulb (*C*), above and below which are two marks (*F*) and (*E*); the volume between these two marks is 20 cu. cm. The neck has a diameter of about 9 mm., and is graduated into tenths of cubic centimeters above the mark (*F*).

METHODS FOR TESTING CEMENT

11. Benzine (62° Baumé naphtha) or kerosene free from water should be used in making the determination.

12. **Method.**—The flask is filled with either of these liquids to the lower mark (*E*), and 64 grams of cement, cooled to the temperature of the liquid, is slowly introduced through the funnel (*B*) (the stem of which should be long enough to extend into the flask to the top of the bulb (*C*)), taking care that the cement does not adhere to the sides of the flask, and that the funnel does not touch the liquid. After all the cement is introduced, the level of the liquid will rise to some division of the graduated neck; this reading, plus 20 cu. cm., is the volume displaced by 64 grams of the cement.

13. The specific gravity is then obtained from the formula

$$\text{Specific gravity} = \frac{\text{Weight of cement, in grams,}}{\text{Displaced volume, in cubic centimeters.}}$$

14. The flask, during the operation, is kept immersed in water in a jar (*A*), in order to avoid variation in the temperature of the liquid in the flask, which should not exceed $\frac{1}{2}$ ° cent. The results of repeated tests should agree within 0.01. The determination of specific gravity should be made on the cement as received; if it should fall below 3.10, a second determination should be made after igniting the sample in a covered dish, preferably of platinum, at a low red heat not exceeding 900° cent. The sample should be heated for 5 minutes with a Bunsen burner (starting with a low flame and gradually increasing to its full height) and then heated for 15 minutes with a blast lamp; the ignition should preferably be made in a muffle.

15. The apparatus may be cleaned in the following manner: The flask is inverted and shaken vertically until the liquid flows freely, and then held in a vertical position until empty; any traces of cement remaining can be removed by pouring into the flask a small quantity of clean liquid benzine or kerosene and repeating the operation.

FINENESS

16. **Significance.**—It is generally accepted that the coarser particles in cement are practically inert, and it is only the extremely fine powder that possesses cementing qualities. The more finely cement is pulverized, other conditions being the same, the more sand it will carry and produce a mortar of a given strength.

17. **Apparatus.**—The fineness of a sample of cement is determined by weighing the residue retained on certain sieves. Those known as No. 100 and No. 200, having approximately 100 and 200

METHODS FOR TESTING CEMENT

wires per linear inch, respectively, should be used. They should be 8 inches in diameter. The frame should be of brass, 8 inches in diameter, and the sieve of brass wire cloth conforming to the following requirements:

No. of sieve.	Diameter of wire.	MESHES PER LINEAR INCH.	
		Warp.	Woof.
100	0.0042 to 0.0048 in.	95 to 101	93 to 103
200	0.0021 to 0.0023 "	192 to 203	190 to 205

The meshes in any smaller space, down to 0.25 inch, should be proportional in number.

18. **Method.**—The test should be made with 50 grams of cement, dried at a temperature of 100° cent. (212° Fahr.).

19. The cement is placed on the No. 200 sieve, which, with pan and cover attached, is held in one hand in a slightly inclined position, and moved forward and backward about 200 times per minute, at the same time striking the side gently, on the up stroke, against the palm of the other hand. The operation is continued until not more than 0.05 gram will pass through in one minute. The residue is weighed, then placed on the No. 100 sieve, and the operation repeated. The work may be expedited by placing in the sieve a few large steel shot, which should be removed before the final one minute of sieving. The sieves should be thoroughly dry and clean.

NORMAL CONSISTENCY

20. **Significance.**—The use of a proper percentage of water in making pastes* and mortars for the various tests is exceedingly important and affects vitally the results obtained.

21. The amount of water, expressed in percentage by weight of the dry cement, required to produce a paste of plasticity desired, termed "normal consistency," should be determined with the Vicat apparatus in the following manner:

22. **Apparatus.**—This consists of a frame (*A*), Fig. 2, bearing a movable rod (*B*), weighing 300 grams, one end (*C*) being 1 cm. in diameter for a distance of 6 cm., the other having a removable needle (*D*), 1 mm. in diameter, 6 cm. long. The rod is reversible,

* The term "paste" is used in this report to designate a mixture of cement and water, and the word "mortar" to designate a mixture of cement, sand, and water.

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and can be held in any desired position by a screw (*E*), and has midway between the ends a mark (*F*) which moves under a scale (graduated to millimeters) attached to the frame (*A*). The paste is held in a conical, hard-rubber ring (*G*), 7 cm. in diameter at the base, 4 cm. high, resting on a glass plate (*H*) about 10 cm. square.

23. **Method.**—In making the determination, the same quantity of cement as will be used subsequently for each batch in making the test pieces, but not less than 500 grams, with a measured quantity

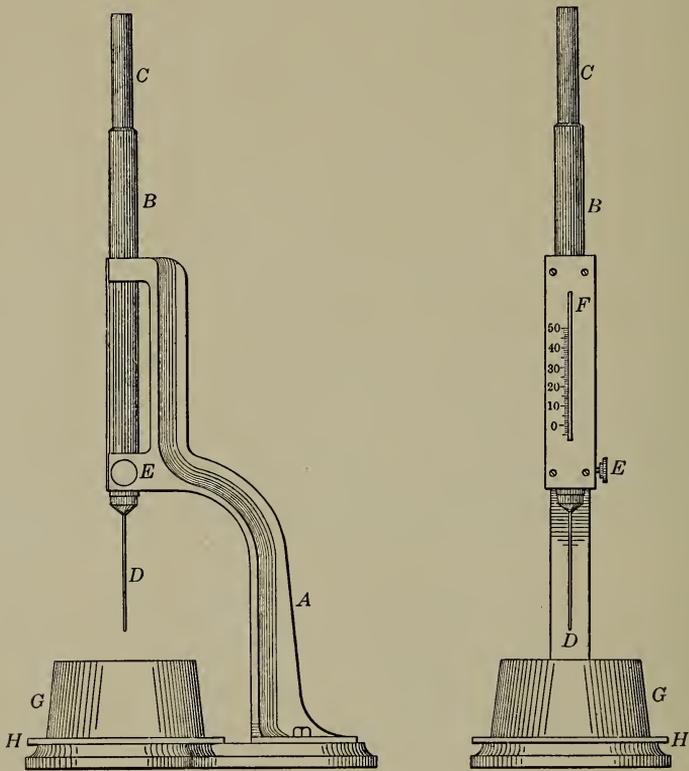


Fig. 2.—Vicat Apparatus

of water, is kneaded into a paste, as described in Paragraph 45, and quickly formed into a ball with the hands, completing the operation by tossing it six times from one hand to the other, maintained about 6 inches apart; the ball resting in the palm of one hand is pressed into the larger end of the rubber ring held in the other hand, completely filling the ring with paste; the excess at the larger end is then removed by a single movement of the palm of the hand; the

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ring is then placed on its larger end on a glass plate and the excess paste at the smaller end is sliced off at the top of the ring by a single oblique stroke of a trowel held at a slight angle with the top of the ring. During these operations care must be taken not to compress the paste. The paste confined in the ring, resting on the plate, is placed under the rod, the larger end of which is brought in contact with the surface of the paste; the scale is then read, and the rod quickly released.

24. The paste is of normal consistency when the cylinder settles to a point 10 mm. below the original surface in one-half minute after being released. The apparatus must be free from all vibrations during the test.

25. Trial pastes are made with varying percentages of water until the normal consistency is obtained.

26. Having determined the percentage of water required to produce a paste of normal consistency, the percentage required for a mortar containing, by weight, one part of cement to three parts of standard Ottawa sand, is obtained from the following table, the amount being a percentage of the combined weight of the cement and sand.

PERCENTAGE OF WATER FOR STANDARD MORTARS

Neat.	One cement, three standard Ottawa sand.	Neat.	One cement, three standard Ottawa sand.	Neat.	One cement, three standard Ottawa sand.
15	8.0	23	9.3	31	10.7
16	8.2	24	9.5	32	10.8
17	8.3	25	9.7	33	11.0
18	8.5	26	9.8	34	11.2
19	8.7	27	10.0	35	11.3
20	8.8	28	10.2	36	11.5
21	9.0	29	10.3	37	11.7
22	9.2	30	10.5	38	11.8

TIME OF SETTING

27. **Significance.**—The object of this test is to determine the time which elapses from the moment water is added until the paste ceases to be plastic (called the “initial set”), and also the time until it acquires a certain degree of hardness (called the “final set” or “hard set”). The former is the more important, since, with the commencement of setting, the process of crystallization begins. As a disturbance of this process may produce a loss of strength, it is desirable to complete the operation of mixing or moulding or incorporating the mortar into the work before the cement begins to set.

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28. **Apparatus.**—The initial and final set should be determined with the Vicat apparatus described in Paragraph 22.

29. **Method.**—A paste of normal consistency is moulded in the hard-rubber ring, as described in Paragraph 23, and placed under the rod (*B*), the smaller end of which is then carefully brought in contact with the surface of the paste, and the rod quickly released.

30. The initial set is said to have occurred when the needle ceases to pass a point 5 mm. above the glass plate; and the final set, when the needle does not sink visibly into the paste.

31. The test pieces should be kept in moist air during the test; this may be accomplished by placing them on a rack over water contained in a pan and covered by a damp cloth; the cloth to be kept from contact with them by means of a wire screen; or they may be stored in a moist box or closet.

32. Care should be taken to keep the needle clean, as the collection of cement on the sides of the needle retards the penetration, while cement on the point may increase the penetration.

33. The time of setting is affected not only by the percentage and temperature of the water used and the amount of kneading the paste receives, but by the temperature and humidity of the air, and its determination is, therefore, only approximate.

STANDARD SANDS

34. The sand to be used should be natural sand from Ottawa, Ill., screened to pass a No. 20 sieve, and retained on a No. 30 sieve. The sieves should be at least 8 in. in diameter; the wire cloth should be of brass wire and should conform to the following requirements:

No. of sieve.	Diameter of wire.	MESHES PER LINEAR INCH.	
		Warp.	Woof.
20	0.016 to 0.017 in.	19.5 to 20.5	19 to 21
30	0.011 to 0.012 "	29.5 to 30.5	28.5 to 31.5

Sand which has passed the No. 20 sieve is standard when not more than 5 grams passes the No. 30 sieve in one minute of continuous sifting of a 500-gram sample.*

* This sand may now (1912) be obtained from the Ottawa Silica Co., at a cost of two cents per pound, f. o. b. cars, Ottawa, Ill.

METHODS FOR TESTING CEMENT

FORM OF TEST PIECES

35. For tensile tests the form of test piece shown in Fig. 3 should be used.

36. For compressive tests, 2-inch cubes should be used.

MOULDS

37. The moulds should be of brass, bronze, or other non-cor-

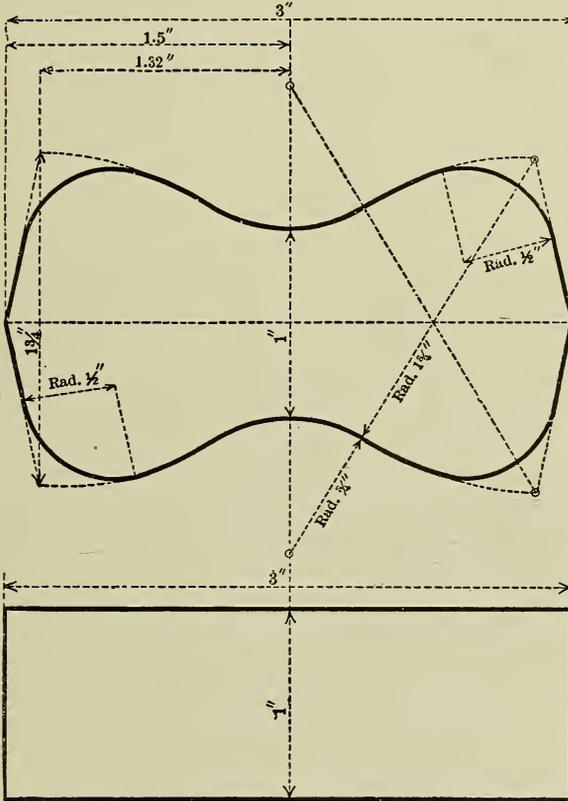


Fig. 3.—Details for Briquette

rodible material, and should have sufficient metal in the sides to prevent spreading during moulding.

38. Moulds may be either single or gang moulds. The latter are preferred by many. If used, the types shown in Figs. 4 and 5 are recommended.

39. The moulds should be wiped with an oily cloth before using.

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MIXING

40. The proportions of sand and cement should be stated by weight; the quantity of water should be stated as a percentage by weight of the dry material.

41. The metric system is recommended because of the convenient relation of the gram and the cubic centimeter.

42. The temperature of the room and of the mixing water should be maintained as nearly as practicable at 21° cent. (70° Fahr.).

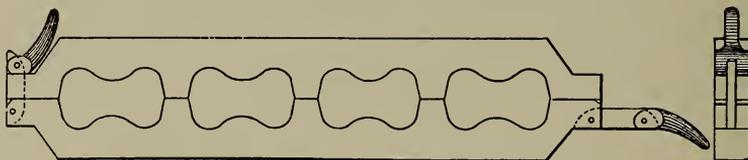


Fig. 4.—Details for Gang Mould

43. The quantity of material to be mixed at one time depends on the number of test pieces to be made; 1000 grams is a convenient quantity to mix by hand methods.

44. The Committee has investigated the various mechanical mixing machines thus far devised, but cannot recommend any of them, for the following reasons: (1) the tendency of most cement is to "ball up" in the machine, thereby preventing working it into a homogeneous paste; (2) there are no means of ascertaining when the mixing is complete without stopping the machine; and (3) it is difficult to keep the machine clean.

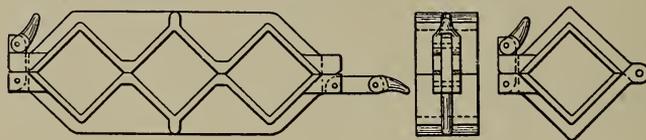


Fig. 5.—Mould for Compression Test Pieces

45. **Method.**—The material is weighed, placed on a non-absorbent surface (preferably plate glass), thoroughly mixed dry if sand be used, and a crater formed in the center, into which the proper percentage of clean water is poured; the material on the outer edge is turned into the center by the aid of a trowel. As soon as the water has been absorbed, which should not require more than one minute, the operation is completed by vigorously kneading

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with the hands for one minute. During the operation the hands should be protected by rubber gloves.

MOULDING

46. The Committee has not been able to secure satisfactory results with existing moulding machines; the operation of machine moulding is very slow; and is not practicable with pastes or mortars containing as large percentages of water as herein recommended.

47. **Method.**—Immediately after mixing, the paste or mortar is placed in the moulds with the hands, pressed in firmly with the fingers, then smoothed off with a trowel without ramming. The material should be heaped above the mould, and, in smoothing off, the trowel should be drawn over the mould in such a manner as to exert a moderate pressure on the material. The mould should then be turned over and the operation of heaping and smoothing off repeated.

48. A check on the uniformity of mixing and moulding may be afforded by weighing the test pieces on removal from the moist closet; test pieces from any sample which vary in weight more than 3 per cent from the average should not be considered.

STORAGE OF THE TEST PIECES

49. During the first 24 hours after moulding, the test pieces should be kept in moist air to prevent drying.

50. Two methods are in common use to prevent drying: (1) covering the test pieces with a damp cloth, and (2) placing them in a moist closet. The use of the damp cloth, as usually carried out, is objectionable, because the cloth may dry out unequally and in consequence the test pieces will not all be subjected to the same degree of moisture. This defect may be remedied to some extent by immersing the edges of the cloth in water; contact between the cloth and the test pieces should be prevented by means of a wire screen or some similar arrangement. A moist closet is so much more effective in securing uniformly moist air, and is so easily devised and so inexpensive, that the use of the damp cloth should be abandoned.

51. A moist closet consists of a soapstone or slate box, or a wooden box lined with metal, the interior surface being covered with felt or broad wicking kept wet, the bottom of the box being kept covered with water. The interior of the box is provided with glass shelves on which to place the test pieces, the shelves being so arranged that they may be withdrawn readily.

52. After 24 hours in moist air, the pieces to be tested after

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longer periods should be immersed in water in storage tanks or pans made of non-corrodible material.

53. The air and water in the moist closet and the water in the storage tanks should be maintained as nearly as practicable at 21° cent. (70° Fahr.).

TENSILE STRENGTH

54. The tests may be made with any standard machine.

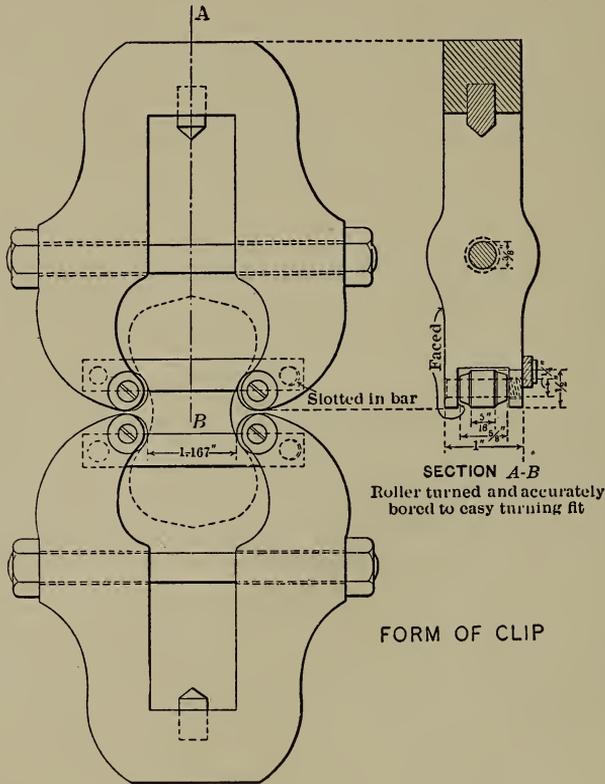


Fig. 6

55. The clip is shown in Fig. 6. It must be made accurately, the pins and rollers turned, and the rollers bored slightly larger than the pins so as to turn easily. There should be a slight clearance at each end of the roller, and the pins should be kept properly lubricated and free from grit. The clips should be used without cushioning at the points of contact.

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56. Test pieces should be broken as soon as they are removed from the water. Care should be observed in centering the test pieces in the testing machine, as cross strains, produced by imperfect centering, tend to lower the breaking strength. The load should not be applied too suddenly, as it may produce vibration, the shock from which often causes the test piece to break before the ultimate strength is reached. The bearing surfaces of the clips and test pieces must be kept free from grains of sand or dirt, which would prevent a good bearing. The load should be applied at the rate of 600 lbs. per min. The average of the results of the test pieces from each sample should be taken as the test of the sample. Test pieces which do not break within $\frac{1}{4}$ in. of the center, or are otherwise manifestly faulty, should be excluded in determining average results.

COMPRESSIVE STRENGTH

57. The tests may be made with any machine provided with means for so applying the load that the line of pressure is along the axis of the test piece. A ball-bearing block for this purpose is shown in Fig. 7. Some appliance should be provided to facilitate placing the axis of the test pieces exactly in line with the center of the ball-bearing.

58. The test piece should be placed in the testing machine, with a piece of heavy blotting paper on each of the crushing faces, which should be those that were in contact with the mould.

CONSTANCY OF VOLUME

59. **Significance.**—The object is to detect those qualities which tend to destroy the strength and durability of a cement. Under normal conditions these defects will in some cases develop quickly, and in other cases may not develop for a considerable time. Since the detection of these destructive qualities before using the cement in construction is essential, tests are made not only under normal conditions but under artificial conditions created to hasten the development of these defects. Tests may, therefore, be divided into two classes: (1) Normal tests, made in either air or water maintained, as nearly as practicable, at 24° cent. (70° Fahr.); and (2) Accelerated tests, made in air, steam or water, at temperatures of 45° cent. (113° Fahr.) and upward. The Committee recommends that these tests be made in the following manner:

60. **Methods.**—Pats, about 3 inches in diameter, $\frac{1}{2}$ in. thick at the center, and tapering to a thin edge, should be made on clean

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glass plates (about 4 in. square) from cement paste of normal consistency, and stored in a moist closet for 24 hours.

61. **Normal Tests.**—After 24 hours in the moist closet, a pat is immersed in water for 28 days and observed at intervals. A similar pat, after 24 hours in the moist closet, is exposed to the air for 28 days or more and observed at intervals.

62. **Accelerated Test.**—After 24 hours in the moist closet, a pat is placed in an atmosphere of steam, upon a wire screen 1 in. above

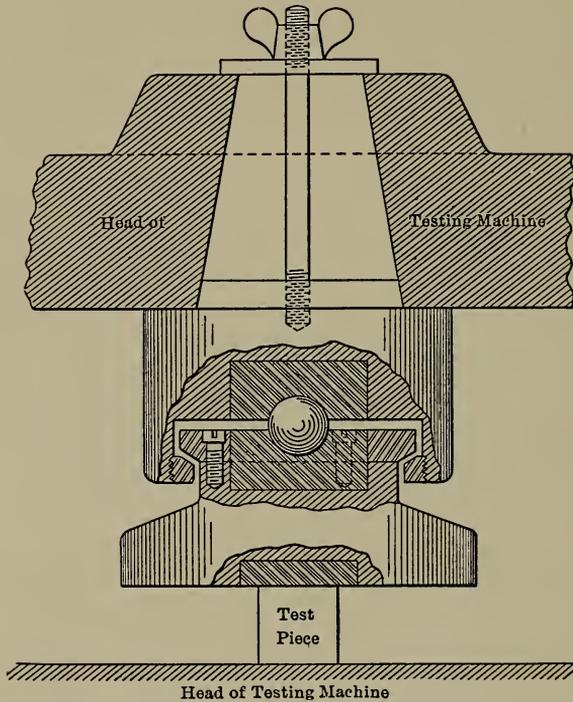


Fig. 7.—Ball-bearing Block for Testing Machine

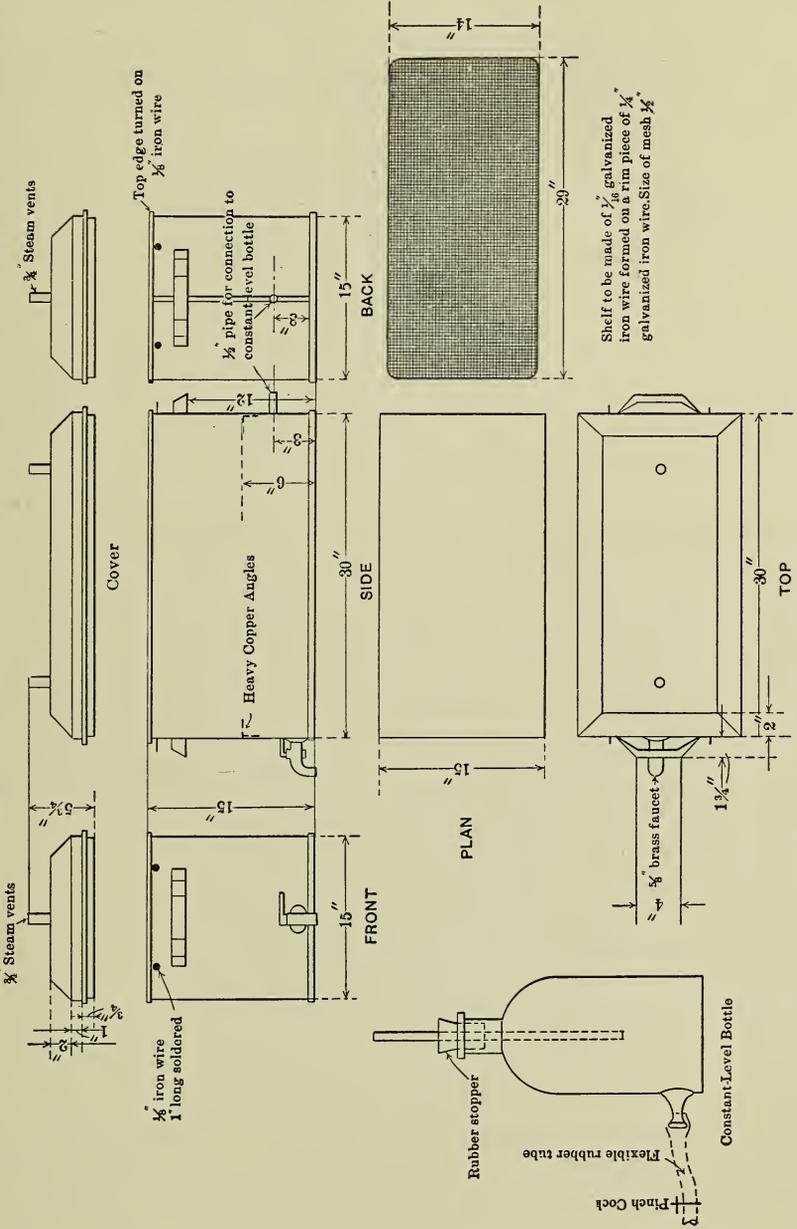
boiling water, for 5 hours. The apparatus should be so constructed that the steam will escape freely and atmospheric pressure be maintained. Since the type of apparatus used has a great influence on the results, the arrangement shown in Fig. 8 is recommended.

63. Pats which remain firm and hard and show no signs of cracking, distortion, or disintegration are said to be "of constant volume" or "sound."

64. Should the pat leave the plate, distortion may be detected

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APPARATUS FOR MAKING ACCELERATED TEST FOR SOUNDNESS OF CEMENT.



To be made of sheet copper weighing 22 oz. per sq. ft., tinned inside.
All seams to be lapped where possible. Hard solder only to be used.

Fig. 8

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best with a straight-edge applied to the surface which was in contact with the plate.

65. In the present state of our knowledge it cannot be said that a cement which fails to pass the accelerated test will prove defective in the work; nor can a cement be considered entirely safe simply because it has passed these tests.

GEORGE S. WEBSTER, *Chairman.*
RICHARD L. HUMPHREY, *Secretary.*
W. B. W. HOWE,
F. H. LEWIS,
S. B. NEWBERRY,
ALFRED NOBLE,
CLIFFORD RICHARDSON,
L. C. SABIN,
GEORGE F. SWAIN.

METHODS FOR TESTING CEMENT*

CONDENSED FOR USE IN SPECIFICATIONS

1. **Sampling.**—Cement in barrels shall be sampled through a hole made in the head, or in one of the staves midway between the heads, by means of an auger or a sampling iron similar to that used by sugar inspectors; if in bags, the sample shall be taken from surface to center. Cement in bins shall be sampled in such a manner as to represent fairly the contents of the bin. The number of samples taken shall be as directed by the Engineer, who will determine whether the samples shall be tested separately or mixed.

The samples shall be passed through a sieve having 20 meshes per linear inch, in order to break up lumps and remove foreign material.

2. **Chemical Analysis.**—The methods to be followed, except for determining the loss on ignition should be those proposed by the Committee on Uniformity in the Analysis of Materials for the Portland Cement Industry, reported in the *Journal* of the Society for Chemical Industry, Vol. 21, p. 12, 1902, and published in *Engineering News*, Vol. 50, p. 60, 1903, and in *Engineering Record*, Vol. 48, p. 49, 1903, and in addition thereto the following:

(a) The insoluble residue may be determined as follows: To a 1-gram sample of the cement are added 30 cu. cm. of water and 10 cu. cm. of concentrated hydrochloric acid, and then warmed until effervescence ceases, and digested on a steam bath until dissolved. The residue is filtered, washed with hot water, and the filter paper and contents digested on the steam bath in a 5% solution of sodium carbonate. This residue is filtered, washed with hot water, then with hot hydrochloric acid, and finally with hot water, and then ignited at a red heat and weighed. The quantity so obtained is the insoluble residue.

(b) The loss on ignition shall be determined in the following manner: $\frac{1}{2}$ gram of cement is heated in a weighed platinum crucible, with cover, for 5 minutes with a Bunsen burner (starting with a low flame and gradually increasing to its full height) and then heated for 15 minutes with a blast lamp; the difference between the

* Accompanying Final Report of Special Committee on Uniform Tests of Cement, dated January 17th, 1912.

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weight after cooling and the original weight is the loss on ignition. The temperature should not exceed 900° cent., or a low red heat; the ignition should preferably be made in a muffle.

3. **Specific Gravity.**—The determination of specific gravity shall be made with a standardized Le Chatelier apparatus. This consists of a flask (*D*), Fig. 1, page 37, of about 120 cu. cm. capacity, the neck of which is about 20 cm. long; in the middle of this neck is a bulb (*C*), above and below which are two marks (*F*) and (*E*); the volume between these two marks is 20 cu. cm. The neck has a diameter of about 9 mm., and is graduated into tenths of cubic centimeters above the mark (*F*).

Benzine (62° Baumé naphtha) or kerosene free from water shall be used in making the determination. The flask is filled with either of these liquids to the lower mark (*E*) and 64 grams of cement, cooled to the temperature of the liquid, is slowly introduced through the funnel (*B*) (the stem of which should be long enough to extend into the flask to the top of the bulb (*C*), taking care that the cement does not adhere to the sides of the flask, and that the funnel does not touch the liquid. After all the cement is introduced, the level of the liquid will rise to some division of the graduated neck; this reading, plus 20 cu. cm., is the volume displaced by 64 grams of the cement. The specific gravity is obtained from the formula,

$$\text{Specific gravity} = \frac{\text{Weight of cement, in grams,}}{\text{Displaced volume, in cubic centimeters.}}$$

The flask, during the operation, is kept immersed in water in a jar (*A*), in order to avoid variations in the temperature of the liquid in the flask, which should not exceed ½° cent. The results of repeated tests should agree within 0.01.

The determination of specific gravity shall be made on the cement as received; if it should fall below 3.10, a second determination shall be made after igniting the sample at a low red heat. The ignition shall be carried out in the following manner:

The flask, during the operation, is kept immersed in water in a jar (*A*) in order to avoid variations in the temperature of the liquid in the flask, which should not exceed ½° cent. The results of repeated tests should agree within 0.01. The determination of specific gravity should be made on the cement as received; if it should fall below 3.10, a second determination should be made after igniting the sample in a covered dish, preferably of platinum, at a low red heat not exceeding 900° cent. The sample should be heated for 5 minutes with a Bunsen burner (starting with a low flame and gradually increasing to its full height) and then heated for 15 minutes

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with a blast lamp; the ignition should preferably be made in a muffle.

4. **Fineness.**—The fineness shall be determined by weighing the residue retained on No. 100 and No. 200 sieves. The sieves, 8 inches in diameter, shall be of brass wire cloth conforming to the following requirements:

No. of sieve.	Diameter of wire.	MESHES PER LINEAR INCH.	
		Warp.	Woof.
100	0.0042 to 0.0048 in.	93 to 101	93 to 103
200	0.0021 to 0.0023 "	192 to 203	190 to 205

The meshes in any smaller space, down to 0.25 inch, shall be proportional in number.

Fifty grams of cement, dried at a temperature of 100° cent. (212° Fahr.), shall be placed on the No. 200 sieve, which with pan and cover attached, is held in one hand in a slightly inclined position, and moved forward and backward about 200 times per minute, at the same time striking the side gently, on the up stroke, against the palm of the other hand. The operation is continued until not more than 0.05 gram will pass through in one minute. The residue is weighed, then placed on the No. 100 sieve, and the operation repeated. The work may be expedited by placing in the sieve a few large steel shot, which should be removed before the final one minute of sieving. The sieves should be thoroughly dry and clean.

5. **Normal Consistency.**—The amount of water, expressed in percentage by weight of the dry cement, required to produce a paste* of the plasticity desired, termed "normal consistency," shall be determined with the Vicat apparatus:

This consists of a frame (*A*), Fig. 2, page 40, bearing a movable rod (*B*), weighing 300 grams, one end (*C*) being 1 cm. in diameter for a distance of 6 cm., the other having a removable needle (*D*), 1 mm. in diameter, 6 cm. long. The rod is reversible, and can be held in any desired position by a screw (*E*), and has midway between the ends a mark (*F*) which moves under a scale (graduated to millimeters) attached to the frame (*A*). The paste is held in a conical, hard-rubber ring (*G*), 7 cm. in diameter at the base, 4 cm. high, resting on a glass plate (*H*) about 10 cm. square.

* The term "paste" is used in these specifications to designate a mixture of cement and water, and the word "mortar" to designate a mixture of cement, sand, and water.

METHODS FOR TESTING CEMENT

In making the determination of normal consistency, the same quantity of cement as will be used subsequently for each batch in making the test pieces, but not less than 500 grams, together with a measured amount of water, is kneaded into a paste, as described in Section 9, and quickly formed into a ball with the hands, completing the operation by tossing it six times from one hand to the other maintained about 6 in. apart; the ball resting in the palm of one hand is pressed into the larger end of the rubber ring held in the other hand, completely filling the ring with paste; the excess at the larger end is then removed by a single movement of the palm of the hand; the ring is then placed on its larger end on a glass plate and the excess paste at the smaller end is sliced off at the top of the ring by a single oblique stroke of a trowel held at a slight angle with the top of the ring. During these operations care must be taken not to compress the paste. The paste confined in the ring, resting on the plate, is placed under the rod, the larger end of which is carefully brought in contact with the surface of the paste; the scale is then read, and the rod quickly released.

The paste is of normal consistency when the cylinder settles to a point 10 mm. below the original surface in one-half minute after being released. The apparatus must be free from all vibrations during the test.

Trial pastes are made with varying percentages of water until the normal consistency is attained.

Having determined the percentage of water required to produce a paste of normal consistency, the percentage required for a mortar containing, by weight, one part of cement to three parts of standard Ottawa sand, shall be obtained from the following table, the amount being a percentage of the combined weight of the cement and sand.

PERCENTAGE OF WATER FOR STANDARD MORTARS

Neat.	One cement, three standard Ottawa sand.	Neat.	One cement, three standard Ottawa sand.	Neat.	One cement, three standard Ottawa sand.
15	8.0	23	9.3	31	10.7
16	8.2	24	9.5	32	10.8
17	8.3	25	9.7	33	11.0
18	8.5	26	9.8	34	11.2
19	8.7	27	10.0	35	11.3
20	8.8	28	10.2	36	11.5
21	9.0	29	10.3	37	11.7
22	9.2	30	10.5	38	11.8

METHODS FOR TESTING CEMENT

6. **Time of Setting.**—The time of setting shall be determined with the Vicat apparatus in the following manner:

A paste of normal consistency is moulded in the hard-rubber ring, as described in Section 5, and placed under the rod (*B*), the smaller end of which is then carefully brought in contact with the surface of the paste, and the rod quickly released.

The cement is considered to have acquired its initial set when the needle ceases to pass a point 5 mm. above the glass plate; and the final set, when the needle does not sink visibly into the paste.

The test pieces must be kept in moist air during the test.

7. **Standard Sand.**—The sand shall be natural sand from Ottawa, Ill., screened to pass a No. 20 sieve, and retained on a No. 30 sieve.

The sieves shall be at least 8 in. in diameter, and the wire cloth shall be of brass wire and shall conform to the following requirements:

No. of sieve.	Diameter of wire.	MESHES PER LINEAR INCH.	
		Warp.	Woof.
20	0.016 to 0.017 in.	19.5 to 20.5	19 to 21
30	0.011 to 0.012 "	29.5 to 30.5	28.5 to 31.5

Sand which has passed the No. 20 sieve is standard when not more than 5 grams passes the No. 30 sieve in one minute of continuous sifting of a 500-gram sample.*

8. **Form of Test Pieces.**—For tensile tests, the form of test pieces shown in Fig. 3, page 43, shall be used.

For compressive tests, 2-in. cubes shall be used.

9. **Mixing and Moulding.**—The material shall be weighed, placed on a non-absorbent surface, thoroughly mixed dry if sand be used, and a crater formed in the center, into which the proper percentage of clean water shall be poured; the material on the outer edge shall be turned into the center by the aid of a trowel. As soon as the water has been absorbed, the operation of mixing shall be completed by vigorously kneading with the hands for one minute.

Immediately after mixing, the paste or mortar shall be placed in the mould (Figs. 4 and 5, page 44) with the hands, pressed in firmly with the fingers, and smoothed off with a trowel without ramming.

* This sand may now (1912) be obtained from the Ottawa Silica Co., at a cost of two cents per pound, f. o. b. cars, Ottawa, Ill.

The material shall be heaped above the mould, and, in smoothing off, the trowel shall be drawn over the mould in such a manner as to exert a moderate pressure on the material; the mould shall then be turned over and the operation of heaping and smoothing off repeated.

The temperature of the room and of the mixing water shall be maintained as nearly as practicable at 21° cent. (70° Fahr.).

10. Storage of the Test Pieces.—During the first 24 hours after moulding, the test pieces shall be stored in a moist closet. This consists of a box of soapstone or slate, or of wood lined with metal, the interior surface being covered with felt or broad wicking kept wet, the bottom of the box being kept covered with water. The interior of the box is provided with glass shelves on which to place the test pieces, the shelves being so arranged that they may be withdrawn readily.

Test pieces from any sample which vary more than 3% in weight from the average, after removal from the moist closet, shall not be considered in determining strength.

After 24 hours in the moist closet, the pieces to be tested after longer periods shall be immersed in water in storage tanks or pans made of non-corrodible material.

The air and water in the moist closet and the water in the storage tanks shall be maintained, as nearly as practicable, at 21° cent. (70° Fahr.).

11. Tests of Tensile Strength. The tests may be made with any standard machine.

The clip is shown in Fig. 6, page 46. It must be made accurately, the pins and rollers turned, and the rollers bored slightly larger than the pins so as to turn easily. There should be a slight clearance at each end of the roller, and the pins should be kept properly lubricated and free from grit. The clips shall be used without cushioning at the points of contact.

The test pieces shall be broken as soon as they are removed from the water. The load shall be applied at the rate of 600 lbs. per minute.

Test pieces which do not break within $\frac{1}{4}$ in. of the center, or are otherwise manifestly faulty, shall be excluded in determining average results.

12. Tests of Compressive Strength.—The tests may be made with any machine provided with means for so applying the load that the line of pressure is along the axis of the test piece. A ball-bearing block for this purpose is shown in Fig. 7, page 48.

The test pieces as soon as they are removed from the water shall be placed in the testing machine, with a piece of heavy blotting paper on each of the crushing faces, which should be those that were in contact with the mould.

13. **Constancy of Volume.**—The tests of constancy of volume comprise “normal tests,” which are made in air or water, maintained as nearly as practicable at 21° cent. (70° Fahr.), and the “accelerated test,” which is made in steam. These tests shall be made in the following manner:

Pats about 3 in. in diameter, $\frac{1}{2}$ in. thick at the center, and tapering to a thin edge, shall be made on clean glass plates (about 4 in. square) from cement paste of normal consistency, and stored in a moist closet for 24 hours.

Normal Tests.—After 24 hours in the moist closet, a pat is immersed in water and observed at intervals. A similar pat, after 24 hours in the moist closet, is exposed to the air for 28 days or more and observed at intervals. The air and water are maintained, as nearly as practicable, at 21° cent. (70° Fahr.).

Accelerated Test.—After 24 hours in the moist closet, a pat is placed in an atmosphere of steam, upon a wire screen 1 in. above boiling water, for 5 hours, the apparatus being such that the steam will escape freely and atmospheric pressure be maintained. The apparatus is shown in Fig. 8, page 49.

The cement passes these tests when the pats remain firm and hard, with no sign of cracking, distortion, or disintegration.

CHEMICAL ANALYSES

Reprint of Report Authorized by the Committee

NEW YORK SECTION SOCIETY FOR CHEMICAL INDUSTRY

METHOD SUGGESTED FOR THE ANALYSIS OF LIMESTONES, RAW MIXTURES AND PORTLAND CEMENTS BY THE COMMITTEE ON UNIFORMITY IN TECHNICAL ANALYSIS WITH THE ADVICE OF W. F. HILLEBRAND

Solution.—One-half gram of the finely powdered substance is to be weighed out, and, if a limestone or unburned mixture, strongly ignited in a covered platinum crucible over a strong blast for 15 minutes, or longer if the blast is not powerful enough to effect complete conversion to a cement in this time. It is then transferred to an evaporating dish, preferably of platinum for the sake of celerity in evaporation, moistened with enough water to prevent lumping, and 5 to 10 c.c. of strong HCl added and digested with the aid of gentle heat and agitation until solution is complete. Solution may be aided by light pressure with the flattened end of a glass rod.* The solution is then evaporated to dryness, as far as this may be possible on the bath.

Silica (SiO₂).—The residue without further heating is treated at first with 5 to 10 c.c. of strong HCl which is then diluted to half strength or less, or upon the residue may be poured at once a larger volume of acid of half strength. The dish is then covered and digestion allowed to go on for 10 minutes on the bath, after which the solution is filtered and the separated silica washed thoroughly with water. The filtrate is again evaporated to dryness, the residue without further heating taken up with acid and water and the small amount of silica it contains separated on another filter paper. The papers containing the residue are transferred wet to a weighed platinum crucible, dried, ignited, first over a Bunsen burner until the carbon of the filter is completely consumed, and finally over the blast for 15 minutes and checked by a further blasting for 10

* If anything remains undecomposed it should be separated, fused with a little Na₂CO₃, dissolved and added to the original solution. Of course a small amount of separated non-gelatinous silica is not to be mistaken for undecomposed matter.

minutes or to constant weight. The silica, if great accuracy is desired, is treated in the crucible with about 10 c.c. of HF1 and four drops of H_2SO_4 , and evaporated over a low flame to complete dryness. The small residue is finally blasted, for a minute or two, cooled and weighed. The difference between this weight and the weight previously obtained gives the amount of silica.*

Alumina and Iron (Al_2O_3 and Fe_2O_3).—The filtrate, about 250 c.c., from the second evaporation for SiO_2 , is made alkaline with NH_4OH after adding HCl, if need be, to insure a total of 10 to 15 c.c. strong acid, and boiled to expel excess of NH_3 , or until there is but a faint odor of it, and the precipitate iron and aluminum hydrates, after settling, are washed once by decantation and slightly on the filter. Setting aside the filtrate, the precipitate is dissolved in hot dilute HCl, the solution passing into the beaker in which the precipitation was made. The aluminum and iron are then reprecipitated by NH_4OH , boiled and the second precipitate collected and washed on the same filter used in the first instance. The filter paper, with the precipitate, is then placed in a weighed platinum crucible, the paper burned off and the precipitate ignited and finally blasted 5 minutes, with care to prevent reduction, cooled and weighed as $Al_2O_3 + Fe_2O_3$.†

Iron (Fe_2O_3).—The combined iron and aluminum oxides are fused in a platinum crucible at a very low temperature with about 3 to 4 grams of $KHSO_4$, or, better, $NaHSO_4$, the melt taken up with so much dilute H_2SO_4 that there shall be no less than 5 grams absolute acid and enough water to effect solution on heating. The solution is then evaporated and eventually heated till acid fumes come off copiously. After cooling and redissolving in water the small amount of silica is filtered out, weighed and corrected by HF1 and H_2SO_4 .‡ The filtrate is reduced by zinc, or preferably by hydrogen sulphide, boiling out the excess of the latter afterwards while passing CO_2 through the flask, and titrated with permanganate.§ The strength of the permanganate solution should not be greater than 0.0040 gr. Fe_2O_3 per c.c.

* For ordinary control in the plant laboratory this correction may, perhaps, be neglected; the double evaporation never.

† This precipitate contains TiO_2 , P_2O_5 , Mn_3O_4 .

‡ This correction of Al_2O_3 Fe_2O_3 for silica should not be made when the HF1 correction of the main silica has been omitted, unless that silica was obtained by only one evaporation and filtration. After two evaporations and filtrations 1 to 2 mg. of SiO are still to be found with the Al_2O_3 Fe_2O_3 .

§ In this way only is the influence of titanium to be avoided and a correct result obtained for iron.

Lime (CaO).—To the combined filtrate from the $\text{Al}_2\text{O}_3 + \text{Fe}_2\text{O}_3$ precipitate a few drops of NH_4OH are added, and the solution brought to boiling. To the boiling solution 20 c.c. of a saturated solution of ammonium oxalate are added, and the boiling continued until the precipitated CaC_2O_4 assumes a well-defined granular form. It is then allowed to stand for 20 minutes, or until the precipitate has settled, and then filtered and washed. The precipitate and filter are placed wet in a platinum crucible, and the paper burned off over a small flame of a Bunsen burner. It is then ignited, redissolved in HCl , and the solution made up to 100 c.c. with water. Ammonia is added in slight excess, and the liquid is boiled. If a small amount of Al_2O_3 separates this is filtered out, weighed, and the amount added to that found in the first determination, when greater accuracy is desired. The lime is then reprecipitated by ammonium oxalate, allowed to stand until settled, filtered and washed,* weighed as oxide by ignition and blasting in a covered crucible to constant weight, or determined with dilute standard permanganate.†

Magnesia (MgO).—The combined filtrates from the calcium precipitates are acidified with HCl and concentrated on the steam bath to about 150 c.c., 10 c.c. of saturated solution of $\text{Na}(\text{HN}_4)\text{HPO}_4$ are added, and the solution boiled for several minutes. It is then removed from the flame and cooled by placing the beaker in ice water. After cooling, NH_4OH is added drop by drop with constant stirring until the crystalline ammonium-magnesium orthophosphate begins to form, and then in moderate excess, the stirring being continued for several minutes. It is then set aside for several hours in a cool atmosphere and filtered. The precipitate is redissolved in hot dilute HCl , the solution made up to about 100 c.c., 1 c.c. of a saturated solution of $\text{Na}(\text{NH}_4)\text{HPO}_4$ added, and ammonia drop by drop, with constant stirring until the precipitate is again formed as described and the ammonia is in moderate excess. It is then allowed to stand for about 2 hours, when it is filtered on a paper or a Gooch crucible, ignited, cooled and weighed as $\text{Mg}_2\text{P}_2\text{O}_7$.

Alkalies (K_2O and Na_2O).—For the determination of the alkalies, the well-known method of Prof. J. Lawrence Smith is to be followed, either with or without the addition of CaCO_3 with NH_4Cl .

* The volume of wash-water should not be too large; *vide* Hillebrand.

† The accuracy of this method admits of criticism, but its convenience and rapidity demand its insertion.

Anhydrous Sulphuric Acid (SO_3).—One gram of the substance is dissolved in 15 c.c. of HCl, filtered and residue washed thoroughly.*

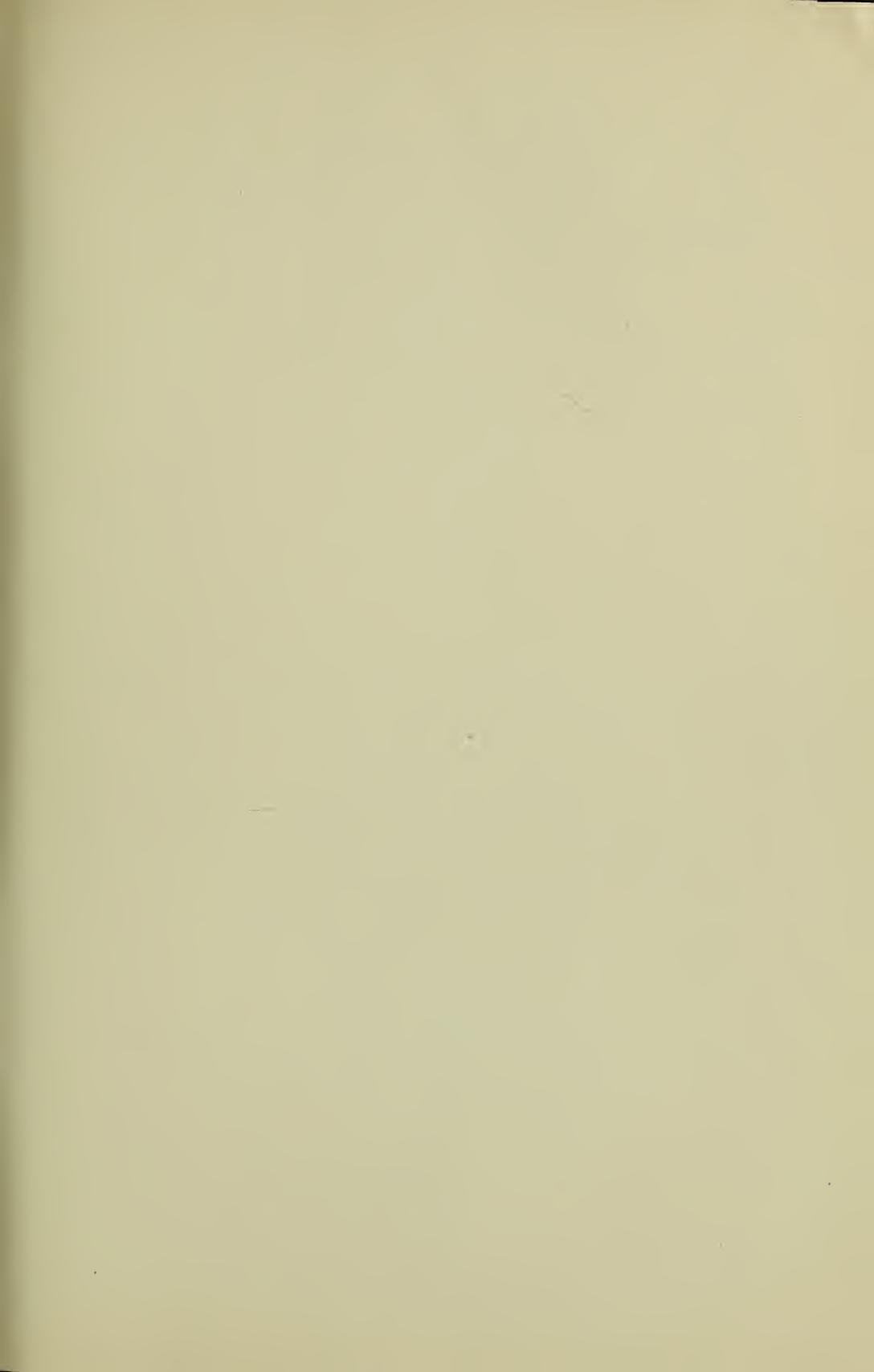
The solution is made up to 250 c.c. in a beaker and boiled. To the boiling solution 10 c.c. of a saturated solution of BaCl_2 is added slowly drop by drop from a pipette and the boiling continued until the precipitate is well formed, or digestion on the steam bath may be substituted for the boiling. It is then set aside over night, or for a few hours, filtered, ignited and weighed as BaSO_4 .

Total Sulphur.—One gram of the material is weighed out in a large platinum crucible and fused with Na_2CO_3 and a little KNO_3 being careful to avoid contamination from sulphur in the gases from source of heat. This may be done by fitting the crucible in a hole in an asbestos board. The melt is treated in the crucible with boiling water and the liquid poured into a tall narrow beaker and more hot water added until the mass is disintegrated. The solution is then filtered. The filtrate contained in a No. 4 beaker is to be acidulated with HCl and made up to 250 c.c. with distilled water, boiled, the sulphur precipitated as BaSO_4 and allowed to stand over night or for a few hours.

Loss on Ignition.—Half a gram of cement is to be weighed out in a platinum crucible, placed in a hole in an asbestos board so that about three-fifths of the crucible projects below, and blasted 15 minutes, preferably with an inclined flame. The loss by weight, which is checked by a second blasting of 5 minutes, is the loss on ignition.

May, 1903: Recent investigations have shown that large errors in results are often due to the use of impure distilled water and reagents. The analyst should, therefore, test his distilled water by evaporation and his reagents by appropriate tests before proceeding with his work.

* Evaporation to dryness is unnecessary, unless gelatinous silica should have separated and should never be performed on a bath heated by gas; *vide* Hillebrand.



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